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FUEL CELL STACKS. (U)

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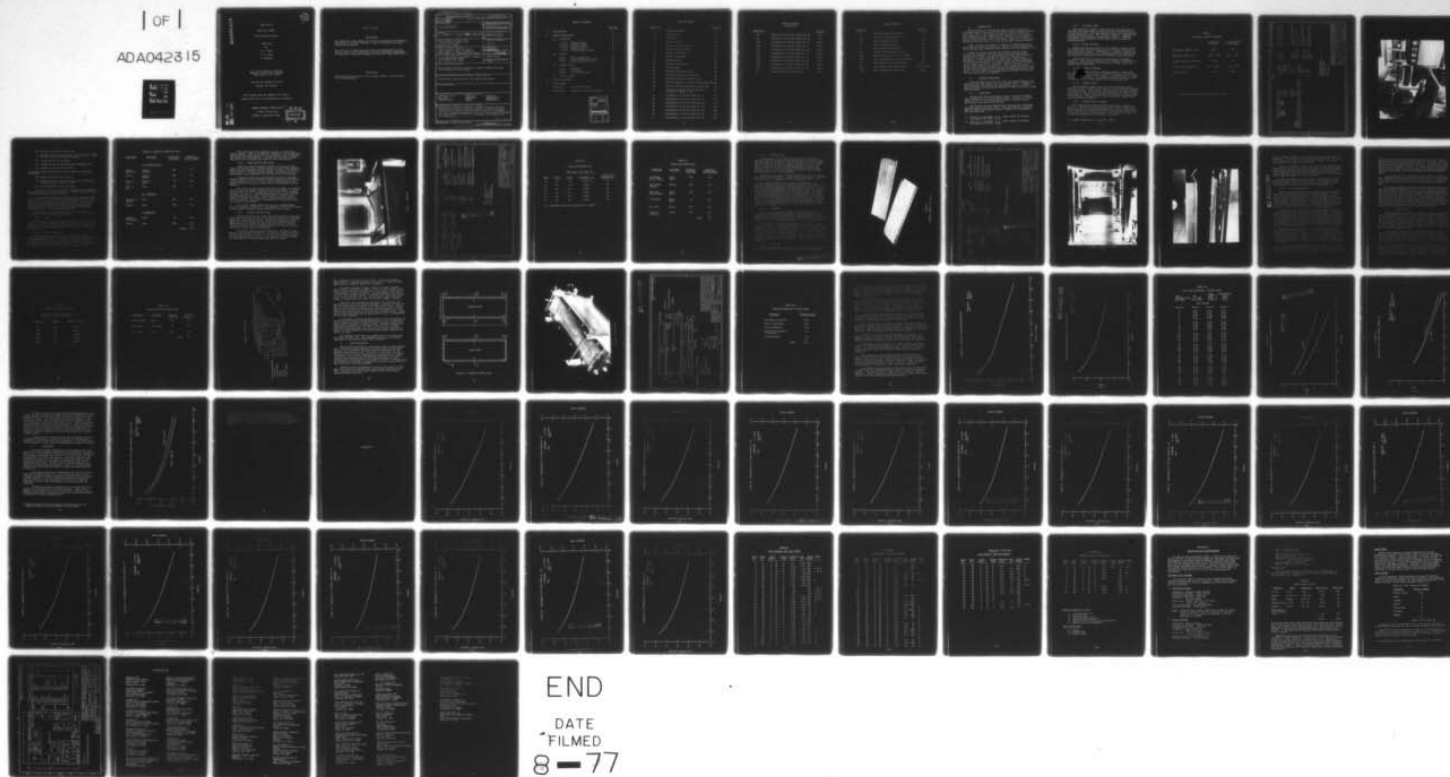
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FUEL CELL STACKS

FINAL TECHNICAL REPORT

JUNE 1977

by

S. G. Abens

B. S. Baker

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U. S. ARMY MOBILITY EQUIPMENT
RESEARCH & DEVELOPMENT COMMAND
Fort Belvoir, VA 22060

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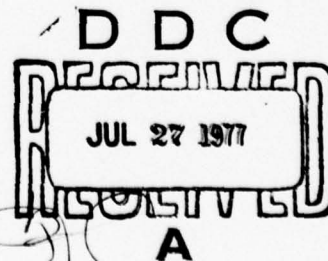
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PHOSPHORIC ACID	SUBSTRATES	GRAPHITE									
FUEL CELL	CATALYSTS	POLARIZATION									
BIPOLAR PLATE	MATRIX	ELECTRODES									
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) <p>Manufacturing methods and techniques for phosphoric acid fuel cell stack production were developed. Electrodes, matrices, and bipolar gas distribution plates for cells with an active area of 0.4 sq. ft. were produced and tested. Assembly and testing of 2-, 10-, and 35-cell stacks was performed. Stacks were operated at 320F for up to 4,000 hours, and tolerance to CO was demonstrated.</p>											

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1. INTRODUCTION

The objective of this program was to develop methods and techniques that can be used in the manufacture of phosphoric acid fuel cell stacks. The effort covered all facets of fuel cell stack manufacture from production of components to the assembly and testing of stacks. The processes evolved on this program utilize technology which is in the public domain or to which the government holds license by virtue of previous contract efforts.

Basic electrode and matrix technology for phosphoric acid fuel cells has been the subject of earlier studies by Energy Research Corporation conducted under contracts to USA MERADCOM.⁽¹⁾⁽²⁾

During the first phase of this program, manufacturing methods for production of electrodes, matrices, and bipolar plates were evolved. At the conclusion of this phase, fifteen 10-cell stacks were assembled (3 stacks were delivered to MERADCOM). In the course of the final phase, thirty additional 10-cell stacks as well as 10 35-cell stacks and 30 experimental 2-cell stacks were built. Of these stacks, 6 10-cell, 3 35-cell, and 5 2-cell stacks were delivered. Also during this phase, process modifications necessitated by the unavailability of some of the original materials for component manufacturing were undertaken. In this report, the manufacturing processes and results of stack evaluation are presented.

2. PROCESS DEVELOPMENT

A prerequisite for a low cost fuel cell stack design is the capability for mass production of the three key cell components - the electrodes, the matrix, and the bipolar plate. In this program, designs and processes amenable to mass production methods were evaluated.

2.1 Electrodes

The ERC acid stack electrodes consist of a thin catalyst layer laminated to a strong support layer. Production of both layers as well as the laminating step can be carried out by conventional mass production methods.

The process has been specifically designed for electrodes with low catalyst loading levels. For this program, a maximum combined anode and cathode catalyst loading of 4 grams/square foot was specified.

(1) Camp, R. N. and Baker, B. S., Final Report on Contract No. DAAK02-71-C-0302, (1972)

(2) Camp, R. N. and Baker, B. S., Final Report on Contract No. DAAK02-72-C-0247, (1973)

2.1.1 Catalyst Layer

The ability to produce a thin distinct catalyst layer is important for delivering a uniform catalyst distribution over the electrode area. The same process is used for the anode and cathode catalyst layers. The catalyst layers are composed of the catalyst and polytetrafluoroethylene binder. Ammonium bicarbonate and an organic liquid (Shell Sol) are used as processing aids.

2.1.1.1 Anode Catalyst

During the initial phases of this program, platinum black was employed as the anode catalyst; the first 10 stacks were built with these anodes. Subsequently, a catalyst composed of platinum black, rhodium black, and tungsten oxide replaced the pure platinum catalyst for better tolerance to CO rich fuels.

The precious metal catalyst is prepared by the Adams' method.⁽³⁾ The catalyst loading for the anode is maintained at 2 grams of precious metal catalyst and 0.7 grams of WO_3 per square foot of electrode.

2.1.1.2 Cathode Catalyst

For the cathode catalyst, platinum black has been used exclusively. In order to insure uniform catalyst distribution, 10% by weight of high purity carbon is also added to the cathode catalyst as an extender. The cathode catalyst loading is also 2 grams of precious metal/square foot of electrode area.

2.1.2 Support Layer

The electrode support layer is a porous graphite paper measuring 0.015 to 0.020 inches thick. In this program, material from two vendors, Union Carbide and Stackpole, has been employed. This support layer combines good conductivity with high porosity (about 85%) to insure low electrode resistance and provide good gas diffusion. Characteristics of the electrode support materials are shown in Table I.

2.1.3 Manufacturing Process

The overall electrode manufacturing process is shown in Figure 1. The process is designed to only include steps which may be carried out with equipment amenable to high rate production. The basic equipment required for the process consists of a rolling mill and a sintering oven. The rolling mill used in the ERC electrode process is shown in Figure 2.

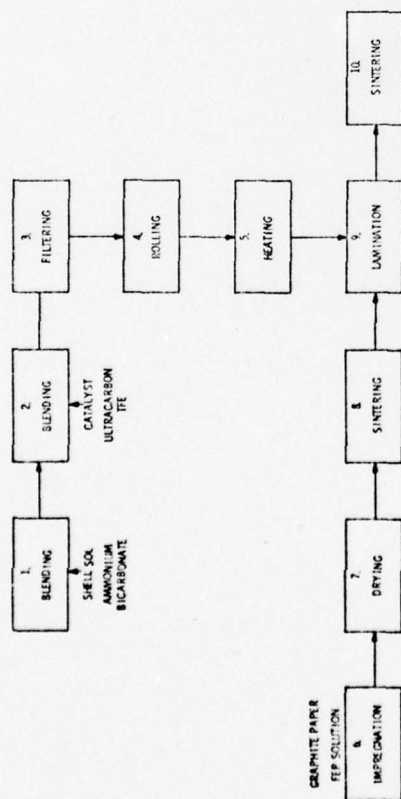
(3) Organic Syntheses, R. Adams Ed. (1932)

TABLE I
ELECTRODE SUPPORT MATERIALS

	STACKPOLE PC 206	UNION CARBIDE VP 0003
Thickness, typical (in.)	.018	.017
Thickness range (in.)	.016 - .020	.015 - .019
Weight, typical (g/sq.ft.)	11.1	7.6
Weight range (g/sq.ft.)	10.6 - 13.5	5.7 - 7.9
Porosity (%)*	84.6	88.8

* Calculated using graphite density of 1.7 g/cm^3

ELECTRODE PROCESS



PROCEDURE
TRANSFER ALL MATERIAL TO BLENDER AND MIX AT "LOW" SETTING FOR 2 MINUTES
TRANSFER ALL MATERIAL TO BLENDER AND MIX AT "LOW" SETTING FOR 2 MINUTES

EQUIPMENT
WATING BLENDOR
MODEL CR-6
WATING BLENDOR
MODEL CR-6

MATERIAL
SHELL SOL 40ml
AMMONIUM BICARBONATE 19g (1)
FROM STEP 1
ADAMS' FLUORIDE 4.3g (2)
ADAMS' BICOXIDE 4.2g (2)
ADAMS' NICOXIDE 1.0g (2)
TUNGSTON OXIDE 1.4g (2)
CATHODE ONLY
PLATING BLACK 7.0g (4)
ULTRACARBON 0.7g (5)
TIE NC 2.4g (6)

PROCEDURE
TRANSFER MATERIAL TO FUNNEL AND FILTER UNTIL FREE OF LIQUID.
ROLL TO SIZE, 11 x 4 1/2 IN.
CUT 6 PIECES, 5 x 15 IN.
HEAT FOR 30 MINUTES IN AIR AT 170°C BETWEEN PAPER
IMMERSE IN BATH FOR 2 MINUTES

EQUIPMENT
BUCHNER FUNNEL #1 D.
ROLLING MILL
C.A.M. I.L. SCIENTISTS
OVEN, NEW ENGLAND OVEN AND
EVIDENCE COMPANY MODEL CASSO
BATH, 20 x 8 x 3 IN.

MATERIAL
FROM STEP 2
FROM STEP 3
FROM STEP 4
GRAPHITE PAPER 5 x 15 IN.
FEP TYPE 12, 1% SOLUTION IN
D.I. WATER

PROCEDURE
SUPPORT WET SHEETS VERTICALLY AND DRY IN AIR FOR 2 HOURS
HEAT FOR 15 MINUTES AT 170°C
PRESS 5 x 15 IN. SHEETS
PLACE IN SINTERING BOX, HEAT IN DOWN AT 1700°C, PLACE
BOX IN PNEUMATIC OVEN AND HOLD AT TEMPERATURE FOR 15
MINUTES (NOTE D)

EQUIPMENT
RACK
OVEN, BLUE M ELECTRIC COM-
PANY, 25.5 FT CONVECTION
0-1500°F
PRESS, 2 X 15 IN. 60 TON
OVEN, BLUE M ELECTRIC COM-
PANY, 25.5 FT CONVECTION
0-1500°F
SINTERING BOX, 1
NITROGEN DRY

MATERIAL
FROM STEP 6
FROM STEP 7
FROM STEP 8
FROM STEP 9
FROM STEP 10

- NOTES:
- ANALYTICAL REAGENT, FISHER SCIENTIFIC CO.
 - ADAMS' PROCESS, ORGANIC SYNTHESIS, R. ADAMS ED. 1930
 - ANALYTICAL REAGENT, WALKERBROT, INC.
 - FUEL CELL GRADE, ENVELOPARD INDUSTRIES
 - UCP-1-M, ULTRA CARBON CORPORATION
 - DUFONT
 - DING, NO. 132116/671
 - 310G FOR ANODE, 300G FOR CATHODE

FIG. 1

ELECTRODE PROCESS

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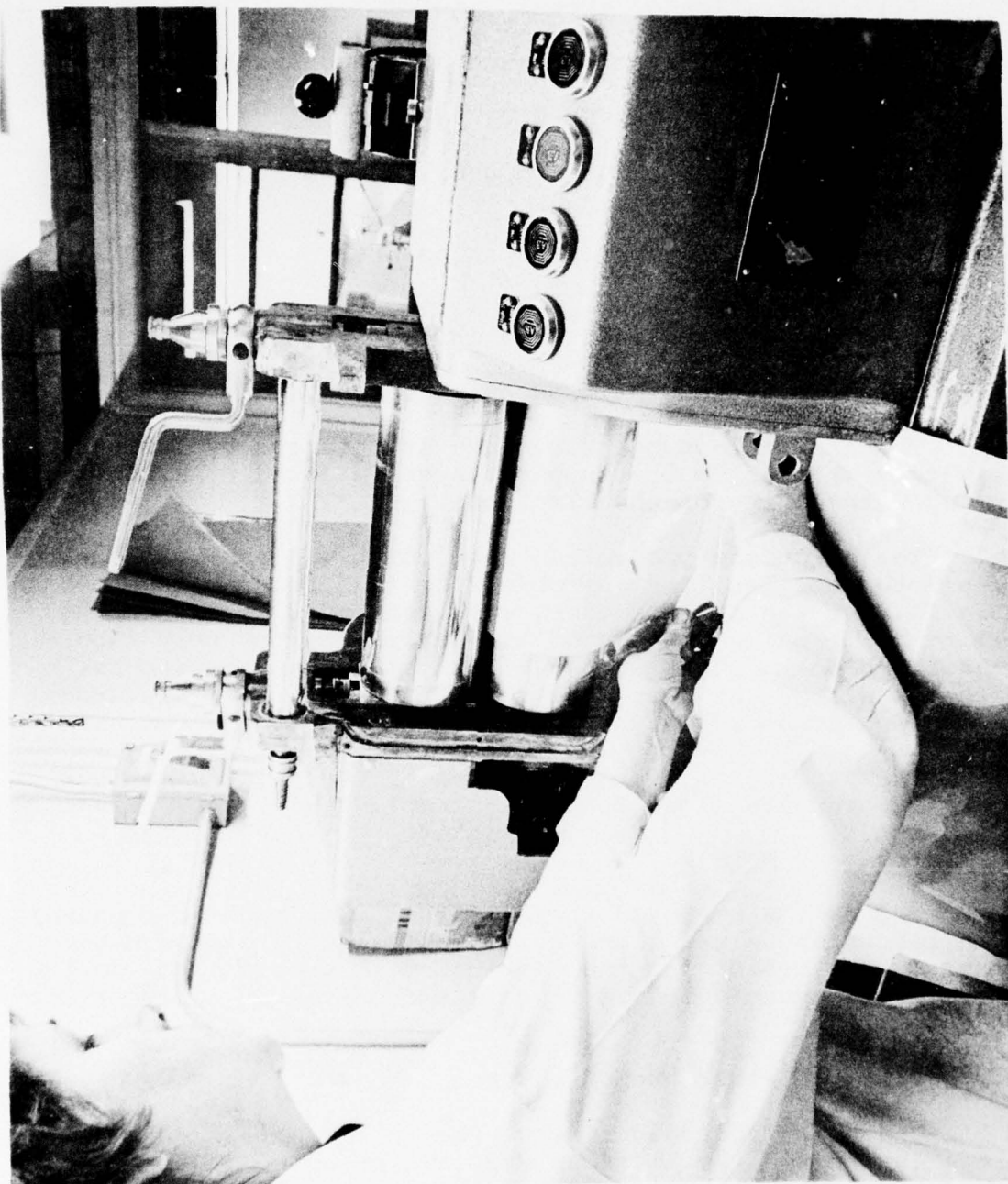


FIGURE 2 ROLLING MILL

The catalyst layer process steps are:

- (1) blending of the catalyst with a rolling medium (Shell Sol) and pore forming agent (NH_4HCO_3),
- (2) filtration of excess liquid,
- (3) rolling to the required size and thickness, and
- (4) removal of the NH_4HCO_3 by heat.

The catalyst layer can be stored until needed for electrode manufacture.

The support layer process steps are:

- (1) immersion in an aqueous dispersion of fluorinated ethylene polymer (FEP 120), and
- (2) air drying followed by sintering.

The final electrode structure is obtained by laminating the catalyst layer to the support layer followed by sintering to develop wetproofing of the electrode.

The electrode production rate which was achievable with our pilot plant facility assembled for this program is shown in Table II. At the present time, catalyst layer sizing on the rolling mill and lamination of two layers are the only labor-intensive process steps in the pilot manufacturing process. These operations, however, can be carried out on continuous process equipment for high production rates.

Acceptance rate for electrodes in the pilot plant was generally over 90% (less than 10% scrap).

2.2 Matrix

The matrix employed in this program is made from phenolic resin fibers and a phenolic resin dispersion by a papermaking process. The resin from the dispersion is precipitated on the fibers in the manufacturing process and serves as a binder for the sheet.

2.2.1 Fiber Preparation

The blown phenolic fibers used in the matrix were initially supplied by American Kynol Corporation (subsidiary of Carborundum Corporation). During the second phase of the program, fibers were obtained from Nippon Kynol Corporation, now the sole source for blown phenolic fibers. The fibers have a diameter of 1-2 μ and an aspect ratio in the order of 100.

TABLE II ELECTRODE PRODUCTION RATE

<u>OPERATION</u>	<u>EQUIPMENT</u>	<u>ELECTRODES/ 8-HR DAY</u>	<u>MANHOURS/ 10-CELL STACK</u>
<u>A. Catalyst Layer</u>			
Blend Materials	Waring Blender	500	0.3
Filter	Buchner Funnel	800	0.2
Roll to Size	Rolling Mill	40	4.0
Heat at 75C	Oven	800	0.2
<u>B. Substrate</u>			
Immerse in FEP 120	Bath	400	0.4
Sinter	Oven	500	0.3
<u>C. Composite</u>			
Laminate at 100 psi	Press	40	4.0
Sinter	Oven	500	0.3
TOTAL			<hr/> 9.7

The fibers are first digested in H_3PO_4 to remove any soluble matter. This is followed by treatment in a conventional laboratory-size fiber beater to insure complete fiber dispersion. The fibers are then digested in 30% KOH solution which etches the smooth fiber surfaces and improves their wettability.

2.2.2 Sheet Molding and Curing

After the fibers have been treated as described above, they are dispersed as an aqueous slurry, and an aqueous phenolic resin emulsion (Monsanto Resinox RI0216) is added. The resin in this emulsion is only partially advanced, and complete cure is accomplished upon subsequent heating during sheet processing.

After the fibers and the emulsion have been thoroughly blended, sufficient 0.1N sulfuric acid is added to lower the pH of the liquid to 4.0 - 4.5. This causes flocculation of the base-stabilized emulsion, and the resin is precipitated on the fibers.

The slurry is now diluted with water to about 0.1% solids concentration and rapidly filtered thru a sheet mold. A photograph of the 18 X 18 in. paper sheet mold used in this program is shown in Figure 3. The resulting sheet is squeezed free of excess liquid and air dried. The final step in the matrix process is sizing the sheet between heated plates in order to bring the matrix to its final thickness and to completely cure the phenolic resin binder.

The various steps needed in the matrix manufacturing process are shown in Figure 4. The quantities of materials shown in this figure produce a sheet measuring 18 X 18 inches.

2.2.3 Process Modifications

Minor modifications to the basic matrix process have been undertaken from time to time in an effort to enhance cell performance and to improve gas seals. These variations are listed in Table III. Some improvement in initial cell performance was demonstrated with matrices having higher porosity. However, the effect of the matrix variables on stack gas seals and life performance appeared to be negligible.

The matrix production rates which were achieved on this program are detailed in Table IV. In general, production yield was over 95% (under 5% scrap). Since the pilot process is one of batch papermaking, we do not visualize any serious problems in adapting it to a conventional papermaking process.

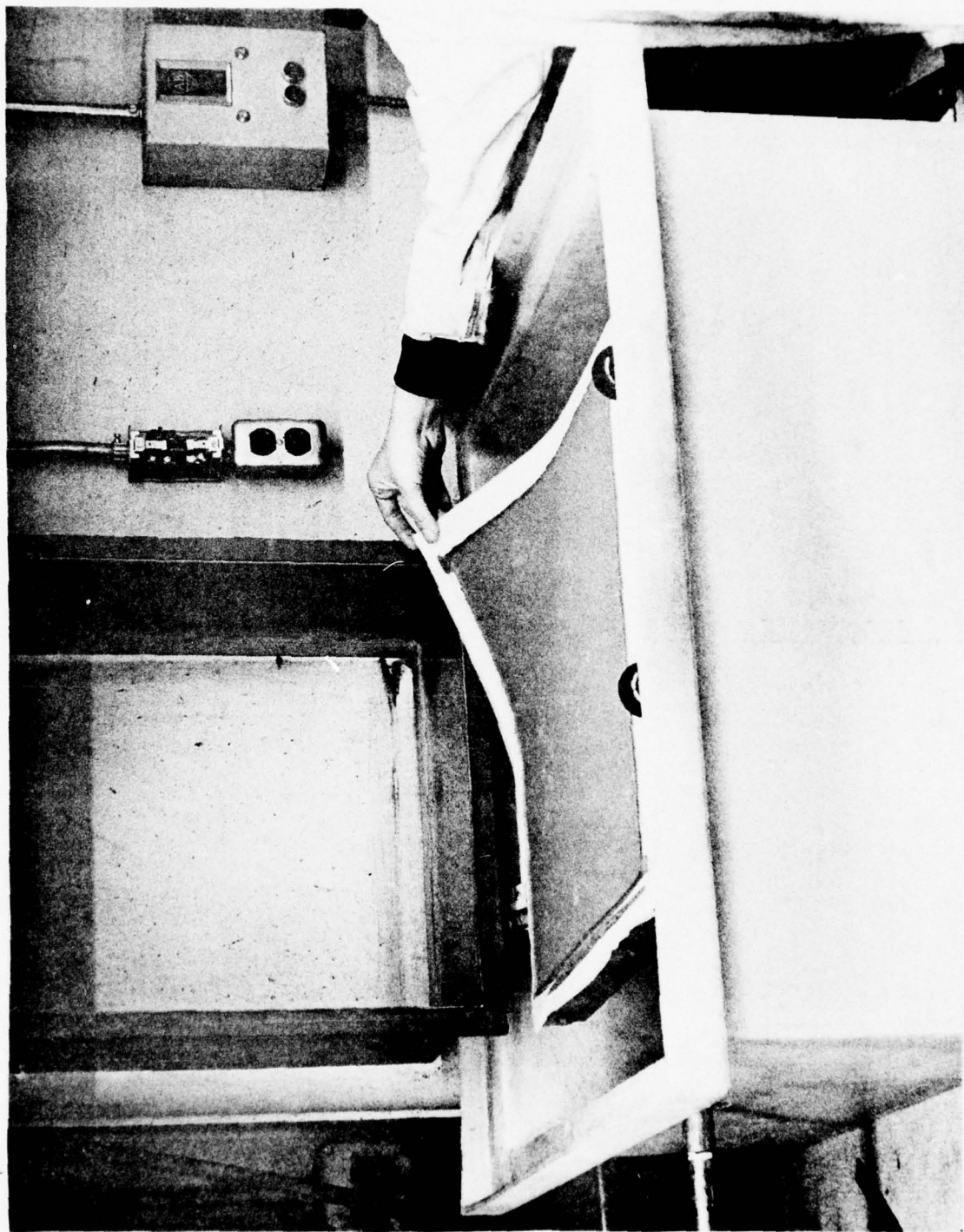


FIGURE 3 SHEET MOLD

10

TABLE III

MATRIX CHARACTERISTICS

MAT'L WT., G/2.2 SQ. FT.

<u>CODE</u>	<u>FIBER</u>	<u>RESIN</u>	<u>THICKNESS, in.</u>	<u>% POROSITY⁽¹⁾</u> <u>(CALCULATED)</u>
A.	28	1.2	0.017	55
B.	42	1.2	0.024	67
D.	42	2.4	0.017	52
E.	42	2.4	0.024	66
H.	42	2.4	0.020	60
I.	35	2.4	0.017	60

(1) Calculated using resin density of 1.2g/cm^3

TABLE IV
MATRIX PRODUCTION RATE

<u>OPERATION</u>	<u>EQUIPMENT</u>	<u>MATRICES/ 8-HR DAY</u>	<u>MANHOURS/ 10-CELL STACK</u>
Acid-treat Kynol fibers	Sheet Mold	100	0.8
Beat Kynol Fibers	Beater	100	0.8
KOH-treat Kynol fibers	Sheet Mold	100	0.8
Form Sheet	Sheet Mold	40	2.0
Wet Press	Press	250	0.2
Press to thickness	Press	250	0.2
TOTAL			4.8

2.3 Bipolar Plate

A photograph of the bipolar plate is shown in Figure 5. The plate is compression molded from a mixture of graphite and thermosetting resin powders. Initially, H-resin from Hercules, Inc. was used as the molding powder, and the first 15 stacks built on this program had plates molded with this material. After termination of production of H-resin was announced by Hercules, Inc., phenol-formaldehyde (PF) resins were employed for production of all bipolar plates.

Conductivity of plates molded with the two types of resin was found to be comparable. With a resin content of 22%, voltage drop thru the plate measured 4mV at 100 amperes/square foot.

The bipolar plate process is shown in Figure 6. The mixture of graphite and resin powders is first leveled to a uniform density in a rectangular mold measuring 5 X 15 inches (the final plate dimensions) and compressed to produce a preform which can be transferred intact to the mold cavity. The mold is mounted in an up-acting hydraulic press as shown in Figure 7. A view of the mold cavity can be seen in Figure 8. The plate is formed by pressing the preform at 150 tons for 6 minutes at 330°F. This is sufficient to cure the resin, and the plate can be ejected at the molding temperature, which obviates the need for any temperature cycling of the mold. The process is completed by heating the plates in an oven which serves to complete curing of the resin.

2.3.1 Graphite

In an effort to improve moldability, modifications in graphite composition were undertaken. Molding art recognizes mixing several types of graphite to improve moldability, i.e., defect-free discharge from the mold. In general, fine and coarse graphites are mixed to provide improved density and strength.

In this work, a number of graphites were tested. A mixture which gives good strength and moldability with PF resins consists of 11 parts of a coarse synthetic graphite (Asbury A-99, average particle size 50 μ) and 4 parts of a fine natural graphite (Asbury 850, average particle size 6 μ). This composition has been found to provide good moldability with resin concentrations over the range of 18 to 37% by weight. Other graphites evaluated included Asbury 230-U (coarse natural), 840, and 870 (both fine natural). No improvement in moldability was obtained with these materials.

2.3.2 PF Resins

All of the PF resins tested on this program were of the two step, Novolac type. Materials included Resinox (Monsanto),

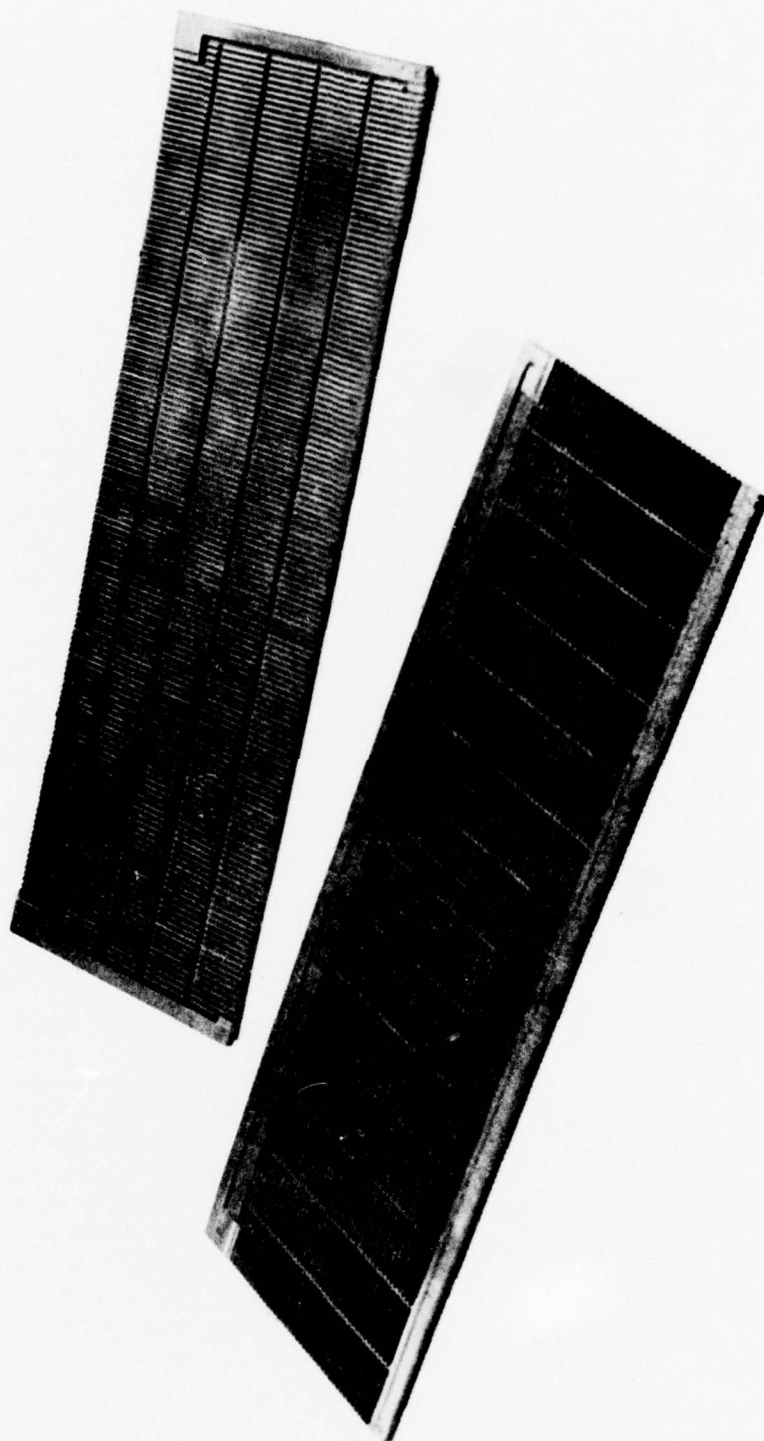
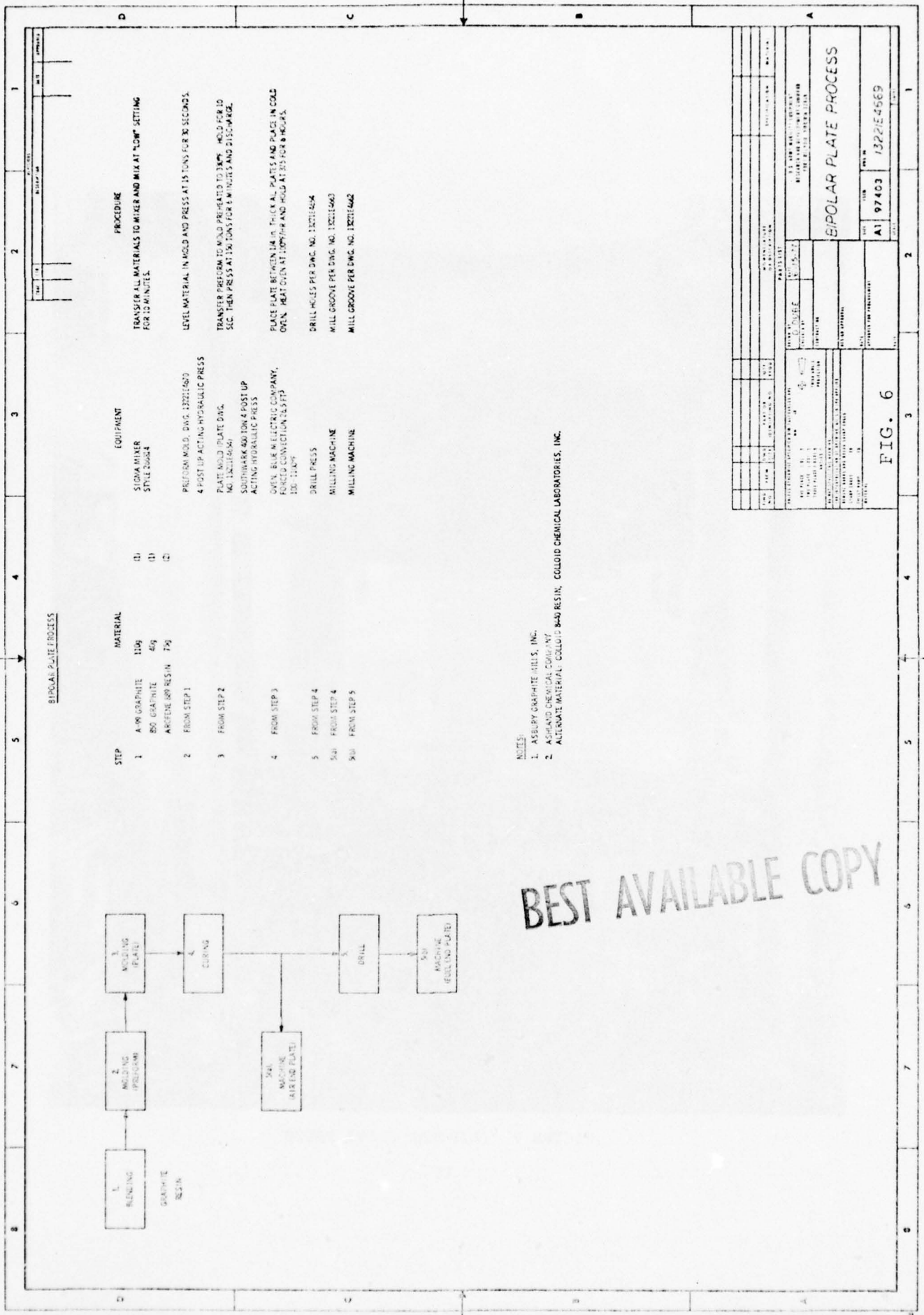


FIGURE 5
BIPOLAR PLATE (5 X 15 in.)



BIPOLAR PLATE PROCESS

STEP	MATERIAL	EQUIPMENT	PROCEDURE
1	(1) A-96 GRAPHITE 112g (1) B-96 GRAPHITE 46g (2) ARFIDE 800 RESIN 72g	SLOW MIXER STYL 26644	TRANSFER ALL MATERIALS TO MIXER AND MIX AT "LOW" SETTING FOR 12 MINUTES.
2	FROM STEP 1	PLASTOM MOLD, DWG. 132216670 4 POST UP ACTING HYDRAULIC PRESS	LEVEL MATERIAL IN MOLD AND PRESS AT 15 TONS FOR 30 SECONDS.
3	FROM STEP 2	PLATE MOLD PLATE DWG. NO. 132216640 SOUTHWARK 400 TON 4 POST UP ACTING HYDRAULIC PRESS	TRANSFER PREFORM TO MOLD PREHEATED TO 340° - HOLD FOR 10 SEC. THEN PRESS AT 150 TONS FOR 6 MINUTES AND DISCHARGE.
4	FROM STEP 3	OUTLY BLUE M ELECTRIC COMPANY, FORCED CONNECTION TESTER 130-11-679	PLACE PLATE BETWEEN 14 IN. THICK PLATES AND PLACE IN COOL OVEN. HEAT OVEN AT 100° F. AND HOLD AT 80° F. FOR 8 HOURS.
5	FROM STEP 4	DRILL PRESS	DRILL HOLES PER DWG. NO. 132216640
5a	FROM STEP 4	MILLING MACHINE	MILL GROOVE PER DWG. NO. 132216640
5b	FROM STEP 5	MILLING MACHINE	MILL GROOVE PER DWG. NO. 132216640

NOTES:

1. ASBURY GRAPHITE MILLS, INC.
2. ASHARD CHEMICAL COMPANY
3. ALTERNATE MATERIAL - COLLOID 8440 RESIN, COLLOID CHEMICAL LABORATORIES, INC.

FIG. 6

PROJECT: 132216640		DATE: 10-15-67	
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APPROVED BY: J. E. HARRIS		DATE: 10-15-67	
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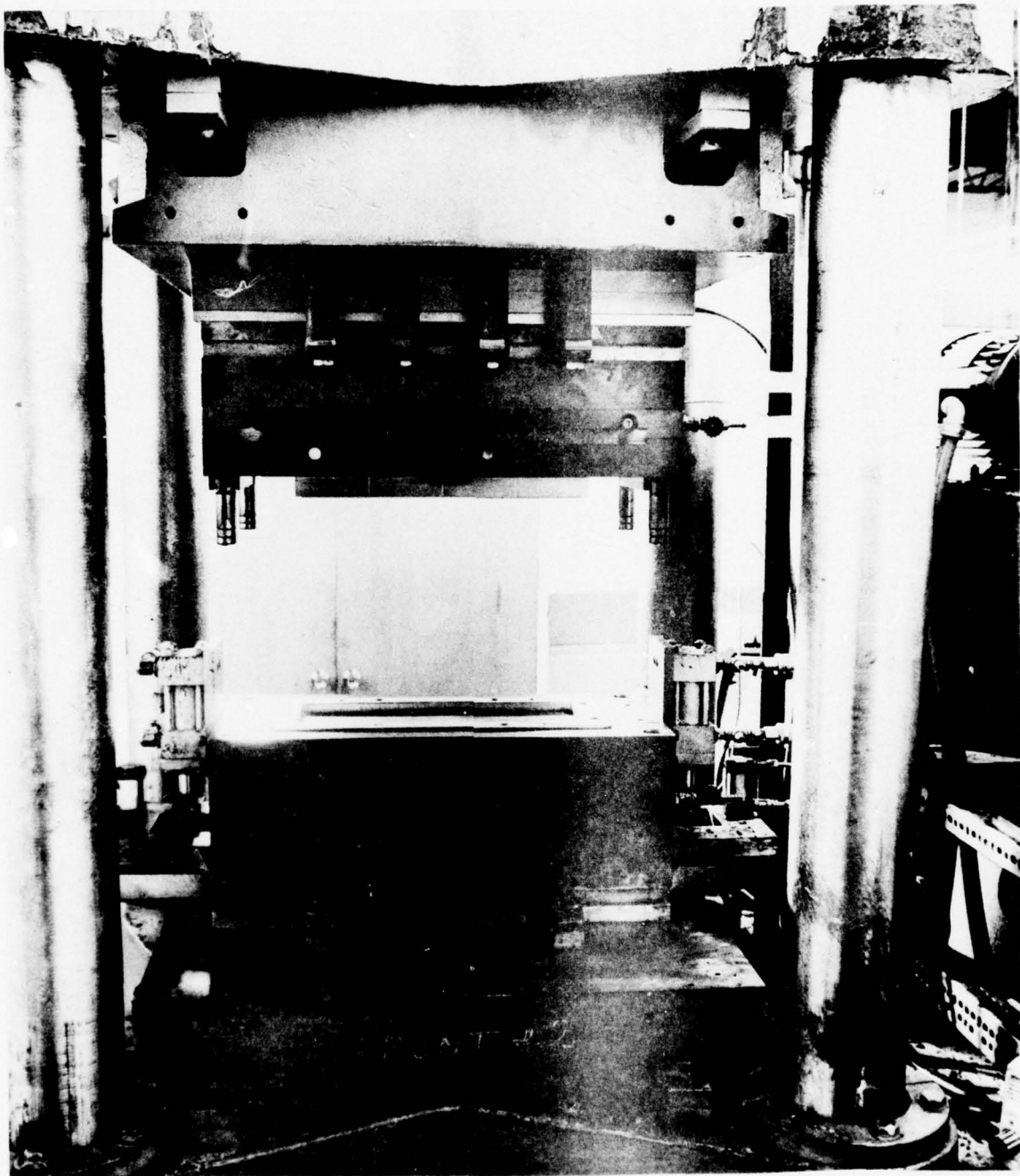


FIGURE 7 BIPOLAR PLATE PRESS

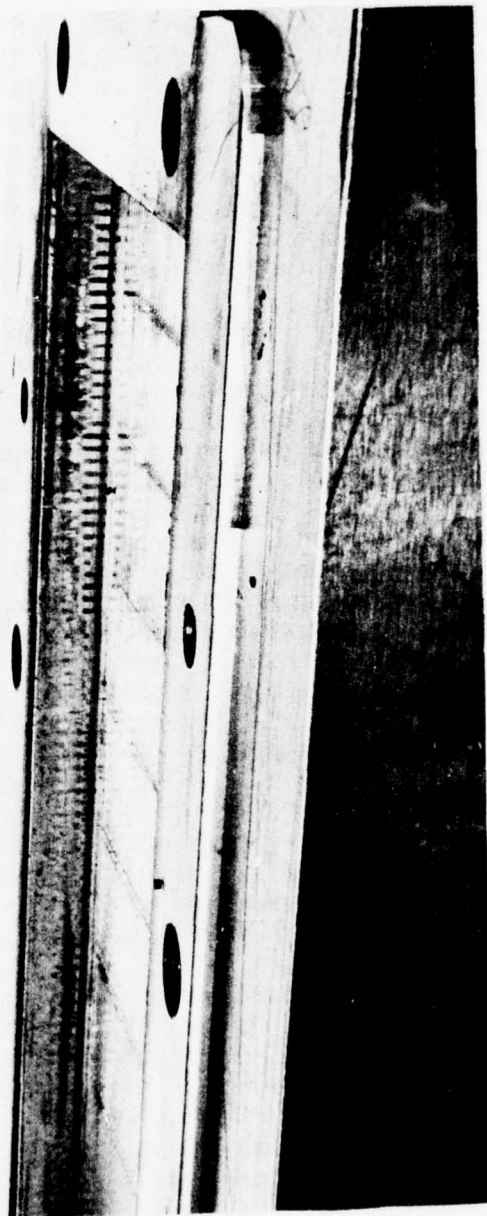
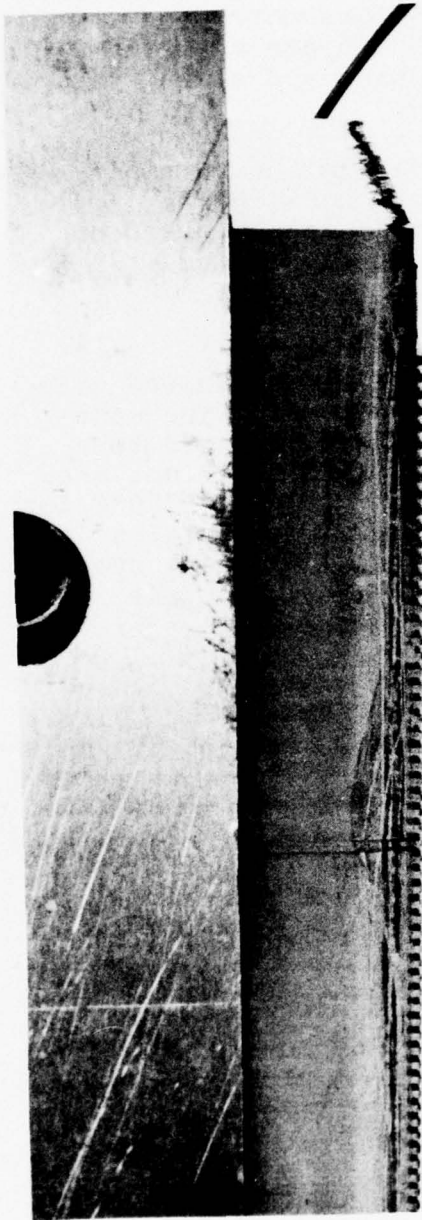


FIGURE 8 BIPOLAR PLATE MOLD

Arofene (Ashland Chemical Co.) and Colloid Resin 8440 (Colloid Chemical). These resins are suitable for molding over the same temperature range that was employed for H-resin, and can be postcured in a few hours at 325-375°F.

Most of the work on the PF resin plate was conducted with Ashland Arofene 890 (now replaced by Ashland with Arofene 889). Additionally, Arofene 882, which contains less unreacted phenol than Arofene 890, and Colloid 8440, which appears to have a coarser particle size than the Arofene resins and gives somewhat better molding properties, were evaluated.

Plates made with all of the above resins showed similar strength, conductivity, and phosphoric acid resistance. The choice of the PF resins has, therefore, been mostly based on moldability rather than on final plate characteristics.

2.3.3 Plate Composition

The initial work with PF resin plates was conducted with compositions containing 18 to 26% by weight resin. The main reason for experimentation over this range was the need to improve yield. Sections broken from these plates did not show any signs of disintegration when immersed in H_3PO_4 at 350°F for several weeks. However, in stacks built with these plates gradual performance degradation was observed. Ten-cell stacks numbered 17 and 20 thru 39, as well as the 35-cell stacks numbered 1 thru 5, had this bipolar plate formulation.

Post mortem inspection of these stacks showed softening and swelling of plate corners around the fill hole. We believe this to be caused by lack of complete densification of the plate corners during molding because of the variable thickness of the plate (the mold comes to a stop on the completely densified web area, which is about 0.050" thick compared to a 0.170" thickness in the corners). The acid is able to penetrate corners because of their porosity and to produce a reaction over the very large surface area of the powdered resin in the resin-graphite matrix.

The obvious approach for correcting the above condition appeared to be densification to the corners by adding more material to the preform in this area, i.e., to mold plates from a "shaped" preform. This approach was pursued with some success; the plate corners did in fact show good resistance to H_3PO_4 in beaker immersion tests. However, molding of plates having consistently acceptable dimensions was found to be difficult by this method.

Another approach to producing plates which are impervious to acid is through the use of high resin content. Plates containing over 30% by weight of resin appear to form corners which are not penetrated by the acid. The limiting condition for this

approach is, of course, the conductivity of the plate. However, as shown in Table V, voltage drops over 20mV at 100ASF, are not reached with resin concentrations below 35%. The figures in this table were obtained by clamping a 2 X 2 in. section of the plate between flat electrodes at 75 psi and measuring the voltage drop thru the plate under a direct current of 100 ampere/square foot. Contact to the plate was made with graphite electrode support paper.

The production rate capability of the pilot process equipment employed on this program is shown in Table VI. A mold cycle time of 12 minutes is indicated for the process; the production rate could, of course, be increased by employing a multicavity mold.

The actual output of plates on this program was not determined by the basic capability of the process, but rather by our ability to produce defect-free plates within desired dimensional tolerance limits. The most common molding defects encountered were missing ribs and blisters; fissures in the web (thin section) of the plate were also encountered. Plates which varied more than 0.003 in. in thickness were also considered unacceptable; this may be an overly tight tolerance for short stacks (10-cells), but may not be too conservative for the taller (35-cell) stacks. In the course of this program, over 3,800 bipolar plates were molded, of which 930 were used in stack construction.

2.4 Stack Assembly

The ERC bipolar acid stack is assembled in the conventional fashion as shown in Figure 9. The stack is assembled with dry matrices, which permits an elastomer adhesive to be applied around the periphery of the matrix for improved gas seals. To fill the stack, 96% H_3PO_4 is wicked by the matrix from the channel which is continuously replenished from an outside reservoir. The stack is maintained at 200-250°F during wicking. By this technique, it is also possible to replenish the electrolyte in the stack at any time during its operating life.

Minor modifications to the basic assembly were undertaken during the course of the program in an effort to improve gas seals. These included 0.002 in. thick Teflon film inserts over the seal area on the air side, and 0.005 in. thick tantalum shims placed over the fuel ribs at the edges of the plate to prevent collapse of the electrode support material into the fuel channels.

Subassemblies are made by glueing the cathode to the matrix and the anode to the plate. The Ta shims are glued to the anode support layer, and the TFE elbows to the exposed cathode plate. The subassemblies are then stacked onto the end support plate, with cement applied to all gas seal surfaces. A fluoroelastomer-based catalyzed cement (C-328 VITON RTV Cement, The Connecticut

TABLE V
VOLTAGE DROP THROUGH PLATE

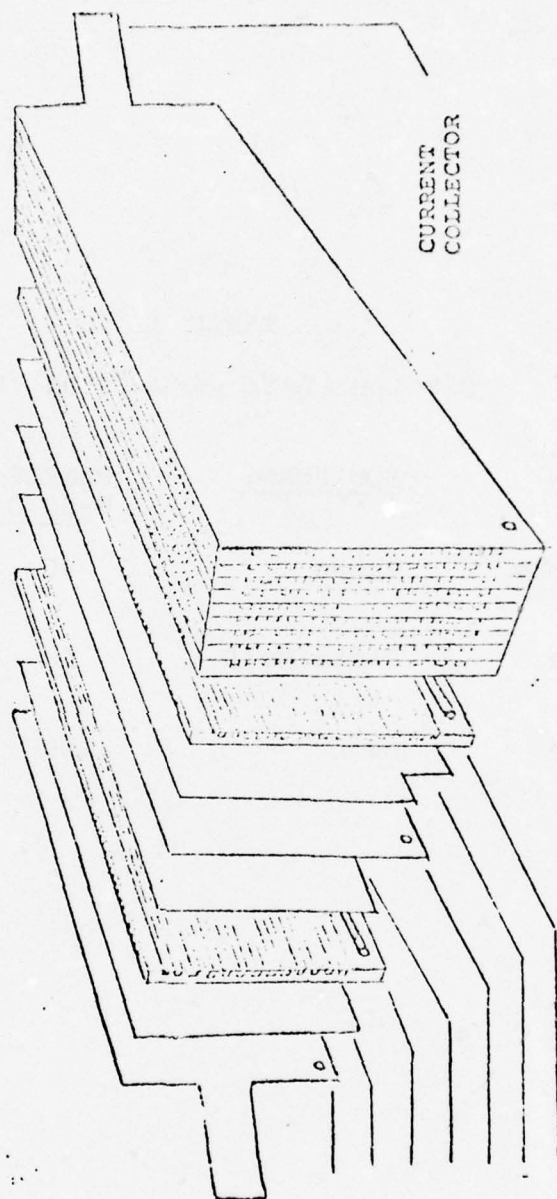
Contact Pressure: 75 lbs sq/in.
Current: 2.7A DC (100ASF)

<u>PLATE NO.</u>	<u>% RESIN</u>	<u>VOLTAGE DROP, mV</u>
558	18	0.004
1641	22	0.006
2569	27	0.008
2491	33	0.018
2285	37	0.032

TABLE VI
BIPOLAR PLATE PRODUCTION RATE

<u>OPERATION</u>	<u>EQUIPMENT</u>	<u>PLATES/ 8-HR DAY</u>	<u>MANHOURS/ STACK</u>
Mold Preform	Press	40	2.2
Mold Plate	Plate Mold	40	2.2
Cure Plate	Oven	200	0.4
TOTAL			4.8

FIGURE 9 STACK ASSEMBLY



22 CURRENT COLLECTOR
 GRAPHITE
 BIPOLAR PLATE
 ANODE
 MATRIX
 CATHODE
 BIPOLAR PLATE

Hard Rubber Co.) is used for all seals. Areas of the matrix where cement is applied are shown in Figure 10. Cement is also applied to the surfaces mating to the matrix.

To prevent passage of gases between the outer graphite plates and the end support plates, a 1/8 in. wide section of the ribs is machined off to the web, and a solid Viton rubber gasket is installed. Current collection is accomplished by the use of 0.005 in. thick silver sheets. To improve the electrical contact to the silver sheets, 0.017 in. thick graphite paper electrode support is placed over the ribs of the outer graphite plates.

The final test assembly of the stack with endplates and gas manifolds (upper air manifold removed) is shown in Figure 11. Most of the stacks had 3/4 in. thick steel outer support plates and 3/4" thick epoxy-fiberglass inner insulating plates. Strip heaters for starting and for maintaining temperature of an idle stack were located between the outer and the inner plates. Steel bars and tie rods were used to supply about 4 tons of compression on the stack assembly.

In an effort to evaluate some materials for lightweight end support plates, panels were assembled with 1/16 in. solid skins and 1" honeycomb cores as shown in Figure 12; both phenolic and aluminum were employed as the materials. Endplates assembled in this manner weighed only 3 1/2 lbs. in the 5 X 15 size, but tended to delaminate when used in stack assemblies, indicating need for a more temperature resistant core-to-skin adhesive (3M AF-III was used).

The manhours needed for the production of a 35-cell stack are shown in Table VII. This estimate is based on continuous operation of the existing pilot plant facility.

3. STACK EVALUATION

The standard procedure in evaluating stacks on this program was to bring the stack temperature to 300°F with the end plate heaters and to determine voltage-current characteristics and hydrogen utilization at 40A. Air flow in these tests was maintained at about 10 times stoichiometric requirement for loads of 40 amperes. Fuel flow to the stack during polarization tests covering the current range of 5-80A was maintained at over 0.8L/min/cell (stoichiometric requirement at 80A is 0.62L/min/cell). The excess fuel was used to insure that sufficient fuel flows to all cells at all current densities.

Typical initial performance of the 10 cell stacks at 300°F was 6.0V at 40A with hydrogen fuel utilization efficiency over 90%. Polarization in the linear region of the V-A plot was typically about 3mV/A/cell.

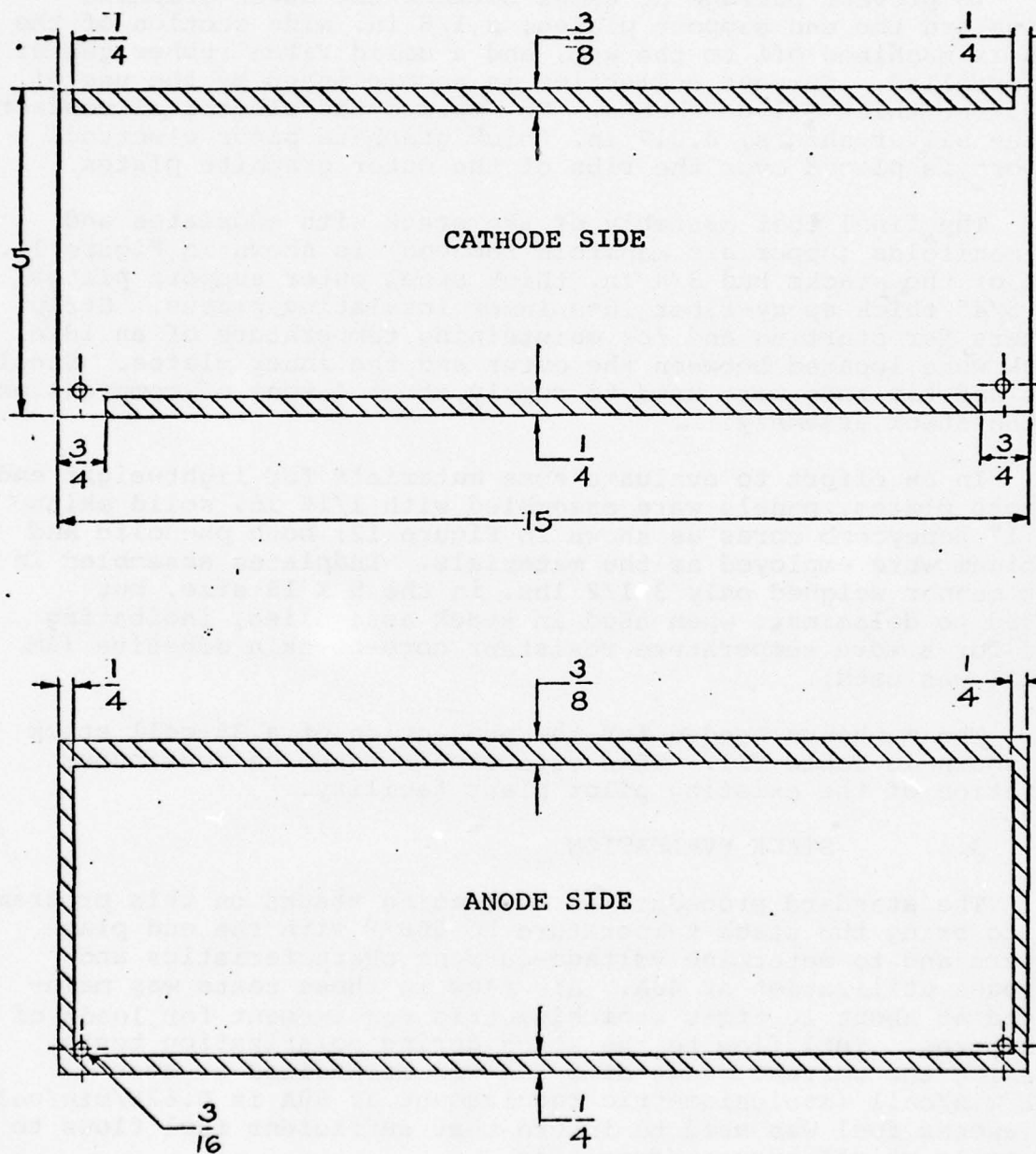


FIGURE 10 CEMENTED MATRIX AREAS

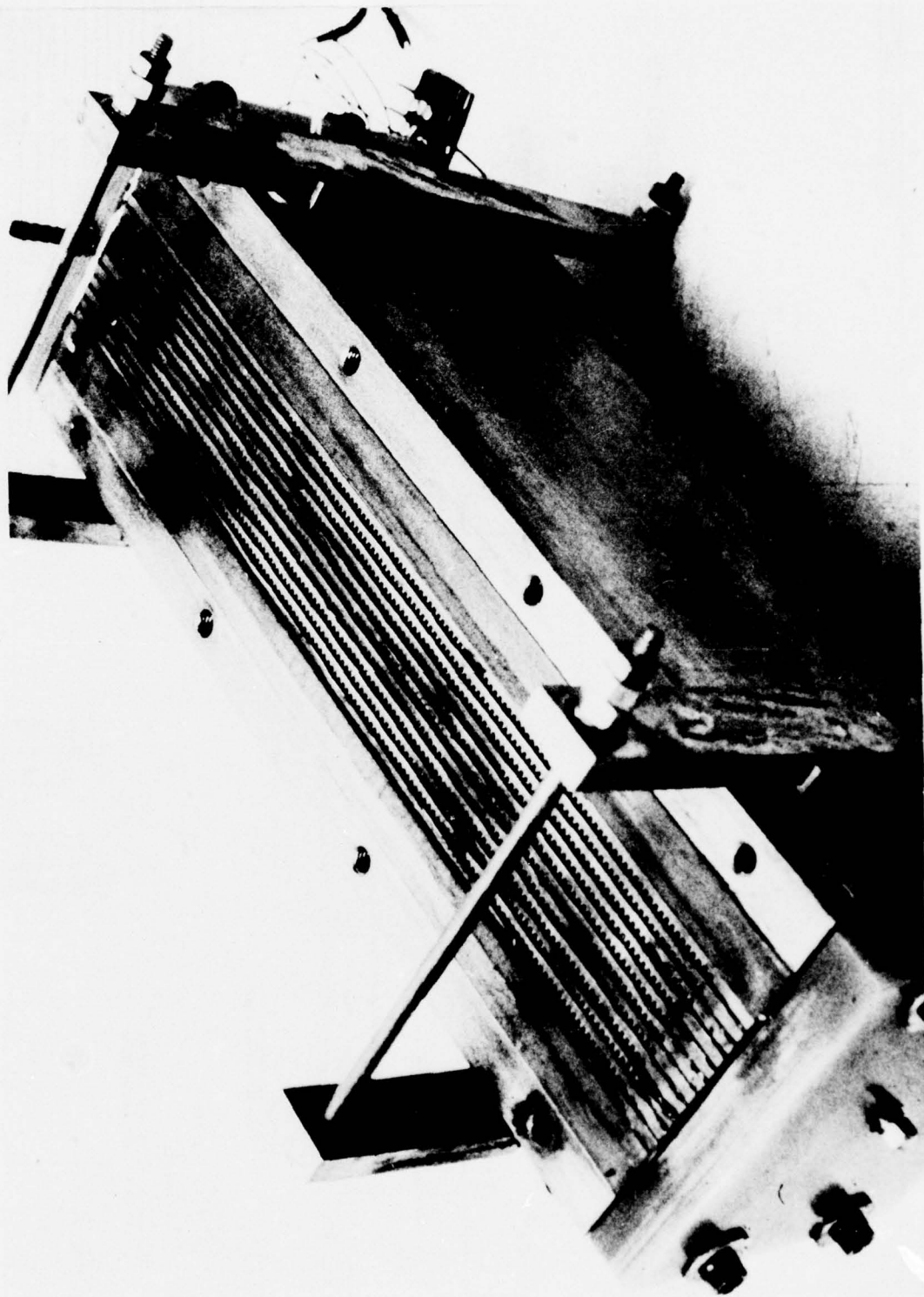


FIGURE 11 TEST ASSEMBLY

FIGURE 12
Honeycomb Endplate
Design

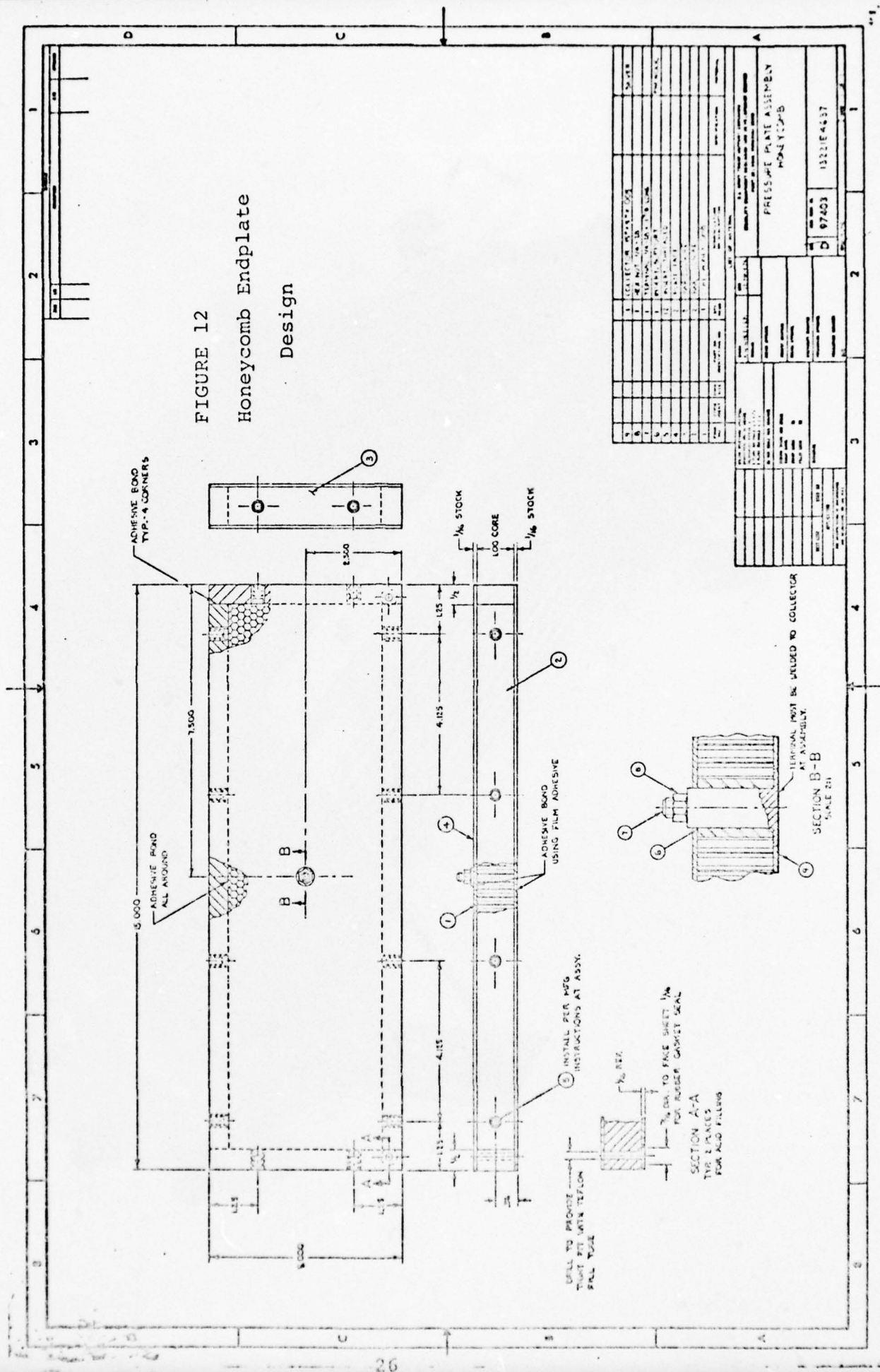


TABLE VII
PRODUCTION RATE FOR 35-CELL STACK

<u>OPERATION</u>	<u>MANHOURS/STACK</u>
Electrode Production	34.0
Matrix Production	16.8
Plate Production	16.8
Auxiliary Components Production	3.5
Stack Assembly	7.0
	<hr/>
TOTAL	78.1

The voltage current plot for a representative 10 cell stack tested under the above conditions is shown in Figure 13. Individual cell voltages were monitored and did not normally show deviations of over 30mV from the average at 40A. The two end cells usually showed the lowest voltage at the somewhat lower temperature of these cells.

Thirty-five cell stack performance was similar (on a per cell basis) to that of 10 cell stacks. A typical polarization curve obtained with these stacks at 300°F with pure hydrogen fuel is shown in Figure 14. Individual cell voltages were also monitored for the 35 cell stacks. Variations between cells did not normally exceed 50mV at 40A. Cell voltages measured for the 35-cell stacks delivered to MERADCOM are shown in Table VIII.

Two-cell stacks generally showed lower cell voltages; this is consistent with the lower voltage observed for end cells in 10 and 35-cell stacks. At the present time, no reason for this reduction in cell voltages has been identified.

After initial characterization, stacks were put on load at 40 amperes. During the first week, the stack was usually operated for up to 8 hours/day and was kept on open circuit for the remaining time. The temperature of the stack was maintained at 250-300°F by the end plate heaters during the off-load period.

Operation with oxygen was attempted on some stacks. In general, a cell voltage increase of 80-100mV was obtained with oxygen compared to air at a current of 40 amperes. The results obtained with this procedure for Stack 10 are shown in Figure 15.

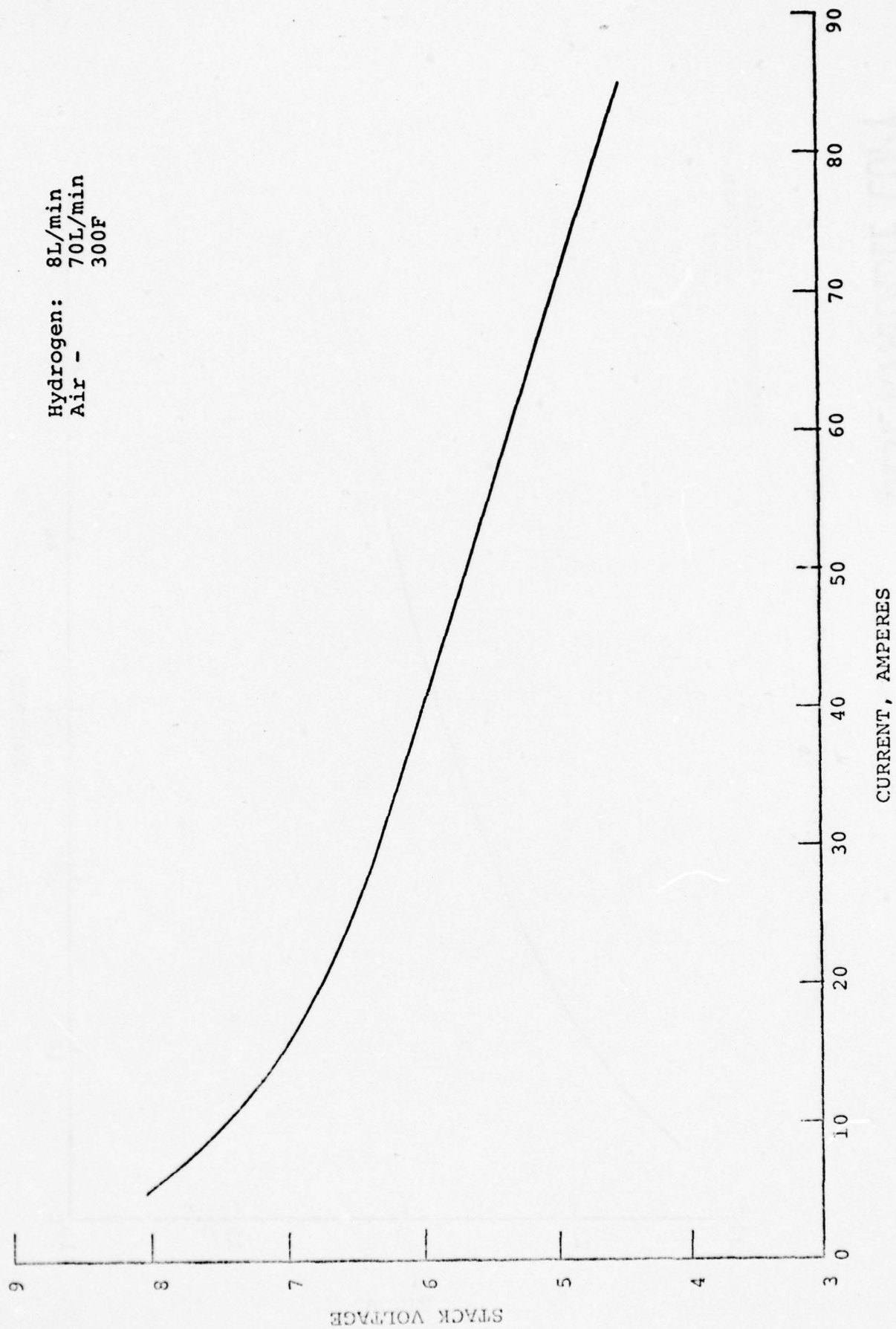
Performance with CO added to the hydrogen fuel was also tested. At 40 amperes, reduction in cell voltage resulting from 2% CO in the fuel was approximately 10mV. Results of a polarization scan for stack 15 with 2% CO in hydrogen are shown in Figure 16.

For stacks built with plates having 20-25% of PF resin, load voltage decay was observed following the first 10 to 50 hours of operation at 40A. Simultaneously, hydrogen utilization in the stack decreased and some cells began to exhibit variation in load voltage with full flow rate variations. This type of stack behavior is typical of a gas cross leak condition.

Disassembly and inspection of these stacks at the conclusion of testing at 40A revealed the plate corner condition described earlier. This appears to have been the major cause of stack degradation. Other degradation mechanisms, such as edge seal leaks, may also have contributed to performance decay.

FIGURE 13 TEN-CELL STACK CHARACTERISTICS

Hydrogen: 8L/min
Air - 70L/min
300F



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FIGURE 14 CHARACTERISTICS of 35-CELL STACK

Hydrogen: 30L/min
Air: 250L/min
300F

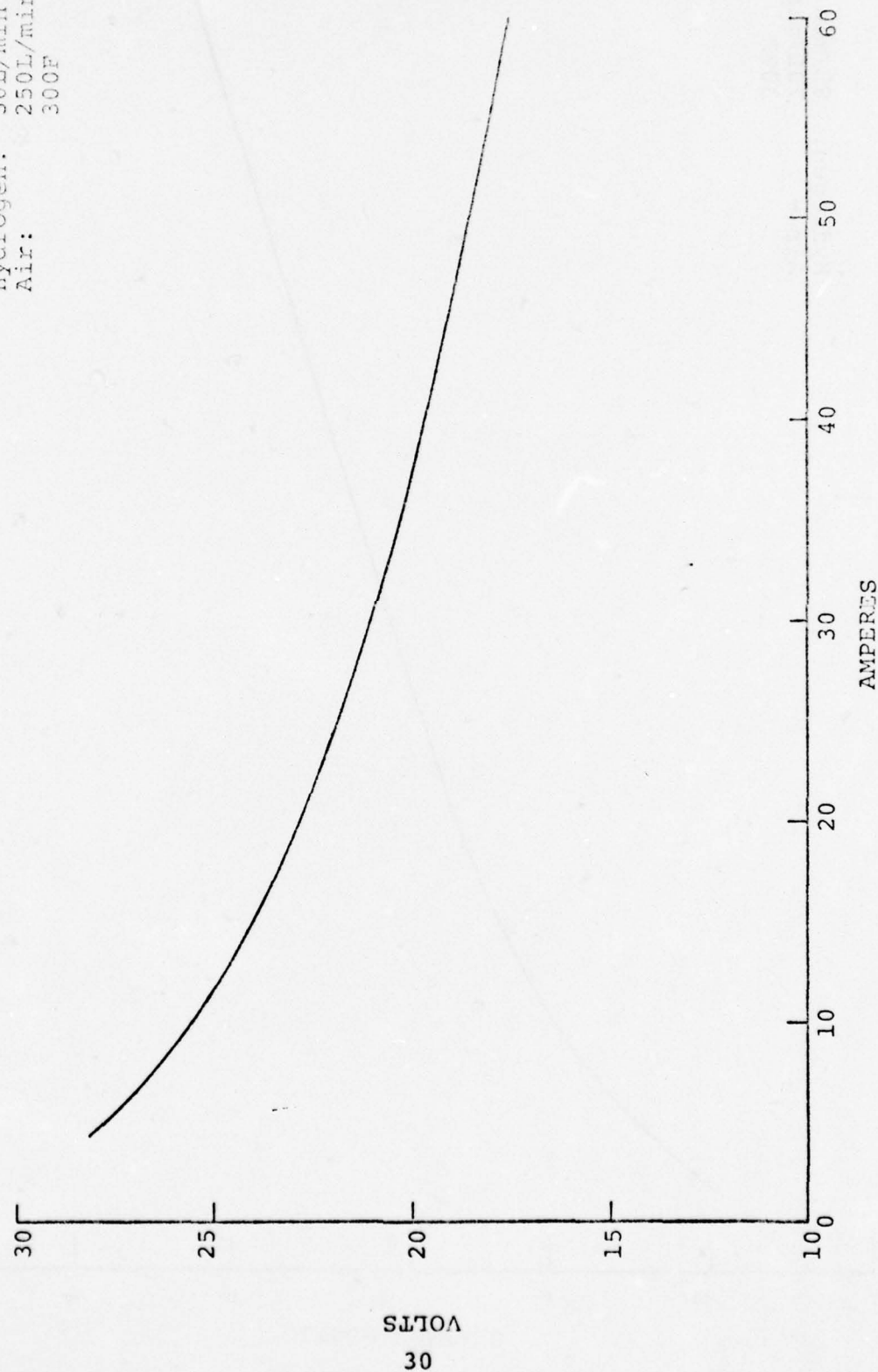


TABLE VIII

CELL LOAD POTENTIALS, 35-CELL STACK

LOAD:	40A	STACK	TEMPERATURE
HYDROGEN FLOW:	30 L/MIN	STACK 8	320°F
AIR FLOW:	250 L/MIN	STACK 9	340°F
		STACK 10	320°F

CELL VOLTAGE

CELL NO.	STACK 8	STACK 9	STACK 10
1	0.56	0.57	0.59
2	0.55	0.58	0.57
3	0.54	0.59	0.57
4	0.58	0.59	0.58
5	0.56	0.60	0.58
6	0.55	0.59	0.58
7	0.53	0.58	0.57
8	0.55	0.60	0.57
9	0.56	0.59	0.57
10	0.57	0.59	0.59
11	0.56	0.59	0.58
12	0.57	0.57	0.58
13	0.55	0.59	0.59
14	0.57	0.57	0.57
15	0.54	0.60	0.59
16	0.57	0.57	0.58
17	0.56	0.57	0.59
18	0.56	0.58	0.56
19	0.55	0.58	0.57
20	0.55	0.56	0.58
21	0.56	0.58	0.58
22	0.53	0.59	0.56
23	0.54	0.57	0.58
24	0.54	0.60	0.59
25	0.51	0.58	0.58
26	0.52	0.60	0.56
27	0.52	0.60	0.57
28	0.51	0.60	0.56
29	0.53	0.57	0.56
30	0.52	0.59	0.59
31	0.51	0.55	0.57
32	0.59	0.59	0.57
33	0.58	0.58	0.58
34	0.59	0.57	0.59
35	0.56	0.57	0.58

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FIGURE 15

EFFECT OF OXYGEN ON LOAD VOLTAGE (STACK 10)

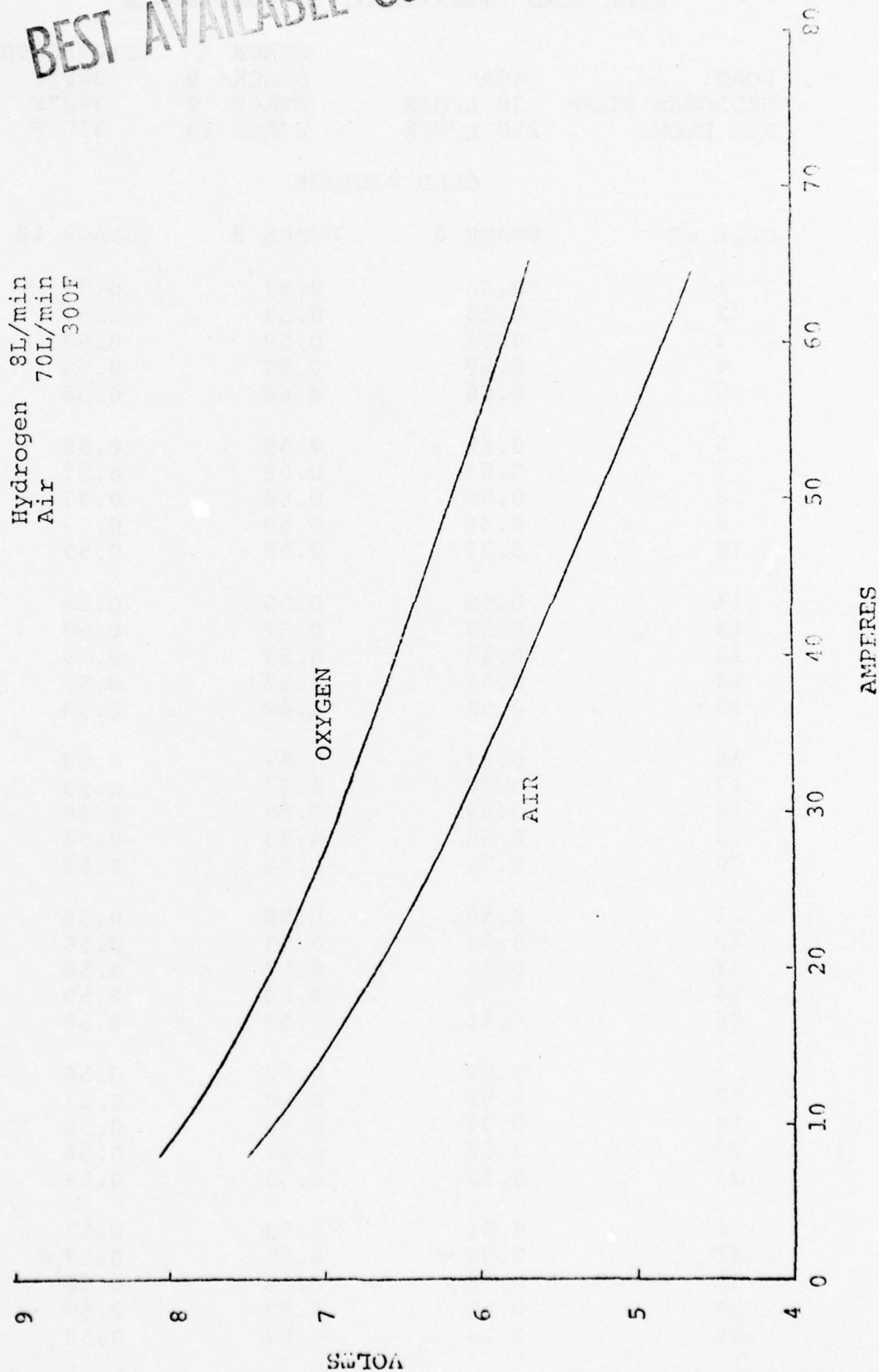
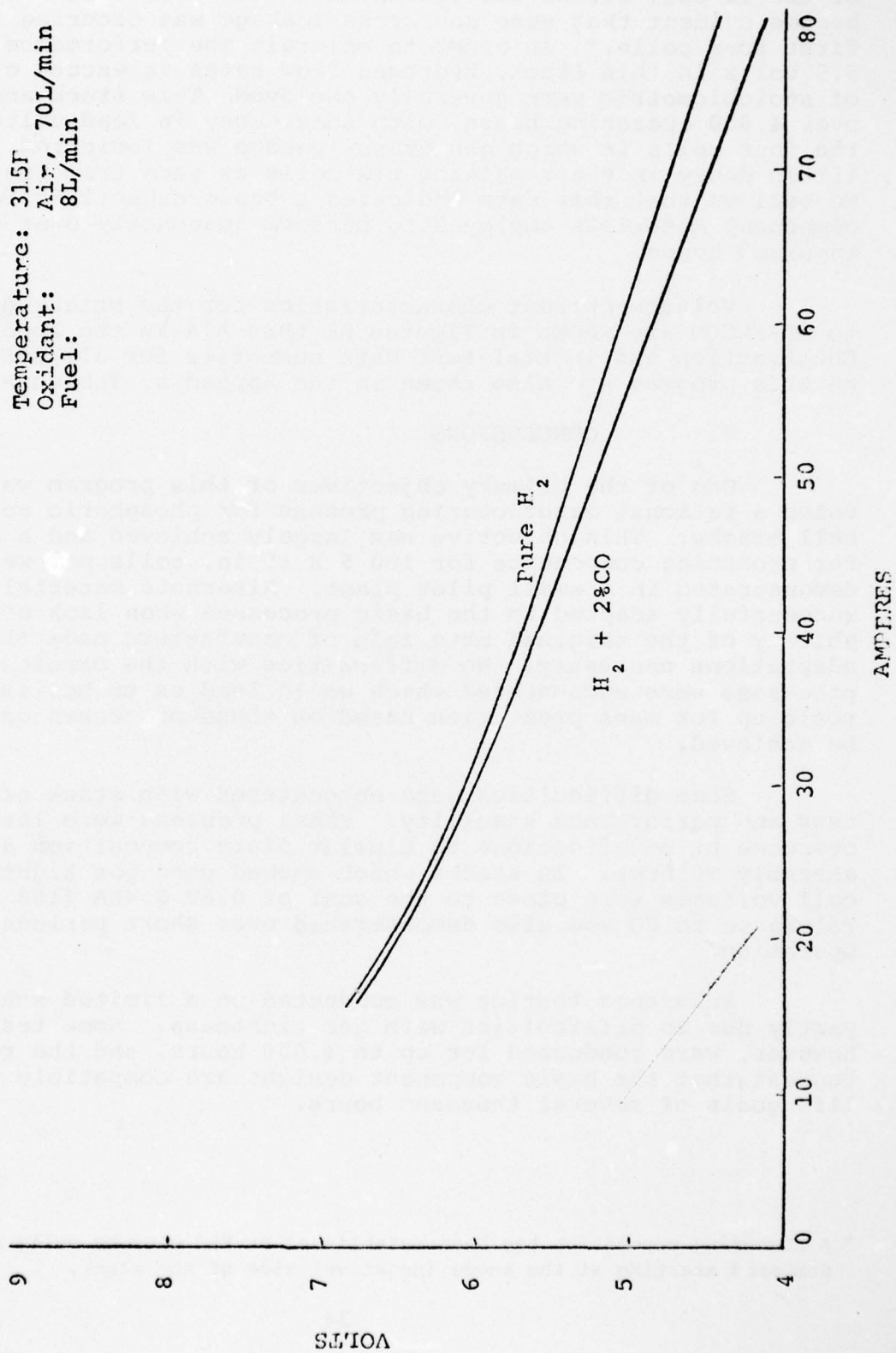


FIGURE 16

EFFECT OF CO ON LOAD VOLTAGE (STACK 15)

Temperature: 315F
Oxidant: Air, 70L/min
Fuel: 8L/min



In order to gain life data with the other components, one of the 10 cell stacks was continued on 40A load even after it had become evident that some gas cross leakage was occurring in the first four cells.* In order to maintain the performance at over 5.5 volts in this stack, hydrogen flow rates in excess of 150% of stoichiometric were generally employed. This stack accumulated over 4,000 operating hours, with some decay in load voltages in the four cells in which gas cross-leakage was indicated, but with little decay in the remaining six cells as seen from Figure 17. We believe that this data indicates a basic capability for all component materials employed to perform adequately over several thousand hours.

Voltage-current characteristics for the stacks delivered to MERADCOM are shown in Figures A1 thru A14 in the Appendix. Construction and initial test data summaries for all stacks built on this program are also shown in the Appendix, Tables A1 thru A3.

4. CONCLUSIONS

One of the primary objectives of this program was to develop a rational manufacturing process for phosphoric acid fuel cell stacks. This objective was largely achieved and a capability for producing components for 100 5 X 15 in. cells per week was demonstrated in a small pilot plant. Alternate materials were successfully adapted to the basic processes when lack of availability of the original materials of manufacture made these adaptations necessary. No difficulties with the manufacturing processes were encountered which would lead us to believe that scale-up for mass production based on these processes could not be achieved.

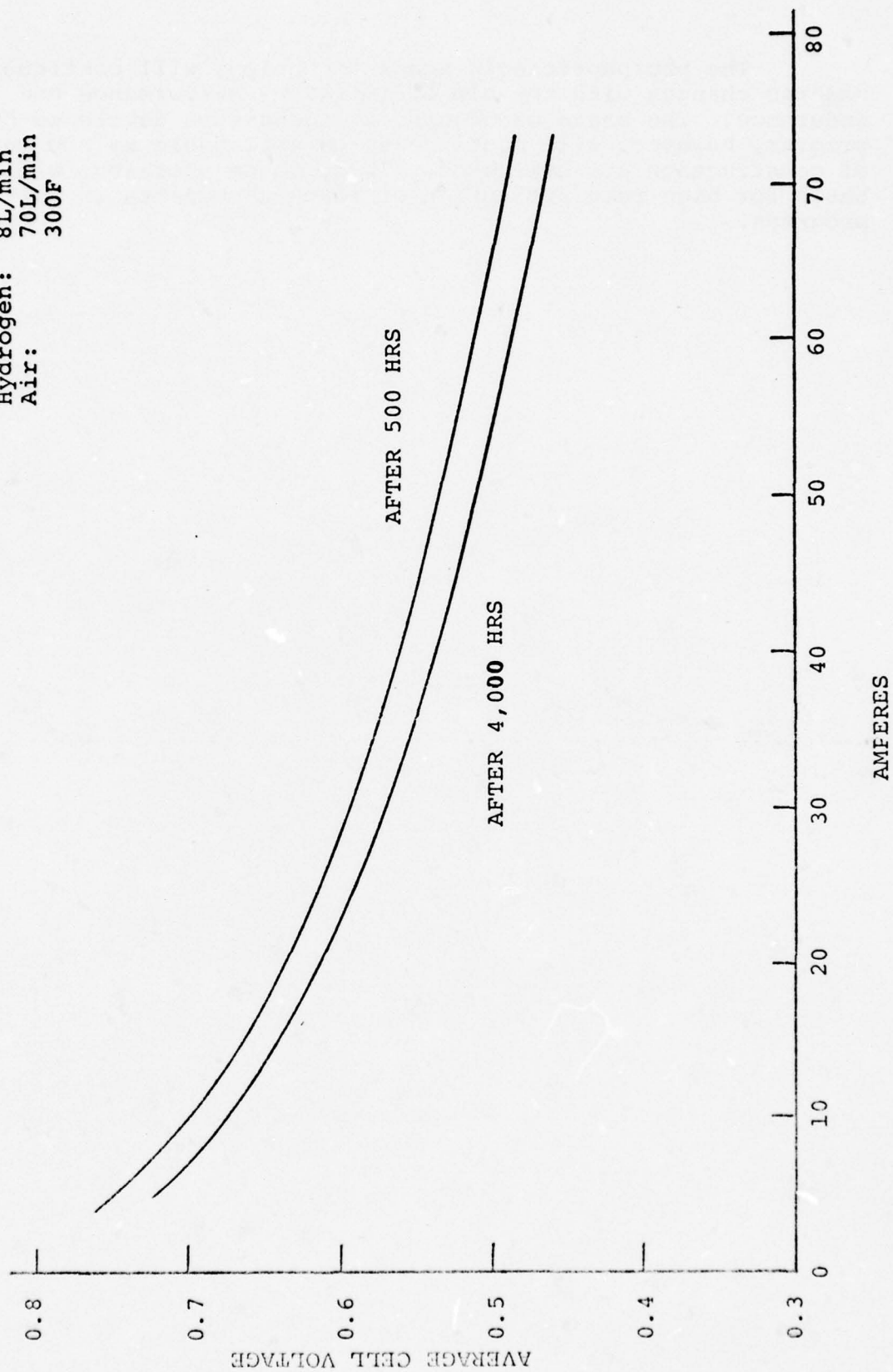
Some difficulties were encountered with stack gas tightness and performance stability. These problems were largely overcome by modifications to bipolar plate composition and stack assembly methods. In stacks which showed good gas tightness, cell voltages were close to the goal of 0.6V @ 40A (100 ASF). Tolerance to CO was also demonstrated over short periods of operation.

Endurance testing was conducted on a limited scale only, partly due to difficulties with gas tightness. Some tests, however, were conducted for up to 4,000 hours, and the results suggest that the basic component designs are compatible with the life goals of several thousand hours.

* A numbering convention has been established at ERC whereby cells are numbered starting at the anode (negative) side of the stack,

FIGURE 17 STACK POLARIZATION AFTER 4,000 HOURS (STACK 17 CELLS 5-10)

Hydrogen: 8L/min
Air: 70L/min
300F



The phosphoric acid stack technology will continue to undergo changes with the aim of improving performance and endurance. The basic manufacturing techniques developed on this program, however, will continue to be applicable as new materials of construction are evaluated. Thus, these processes will form a basis for high rate production of stack components in future programs.

APPENDIX A

FIGURE A1 PERFORMANCE OF 10-CELL STACK NO. 5

HYDROGEN: 8 L/min
AIR: 90 L/min
300°F

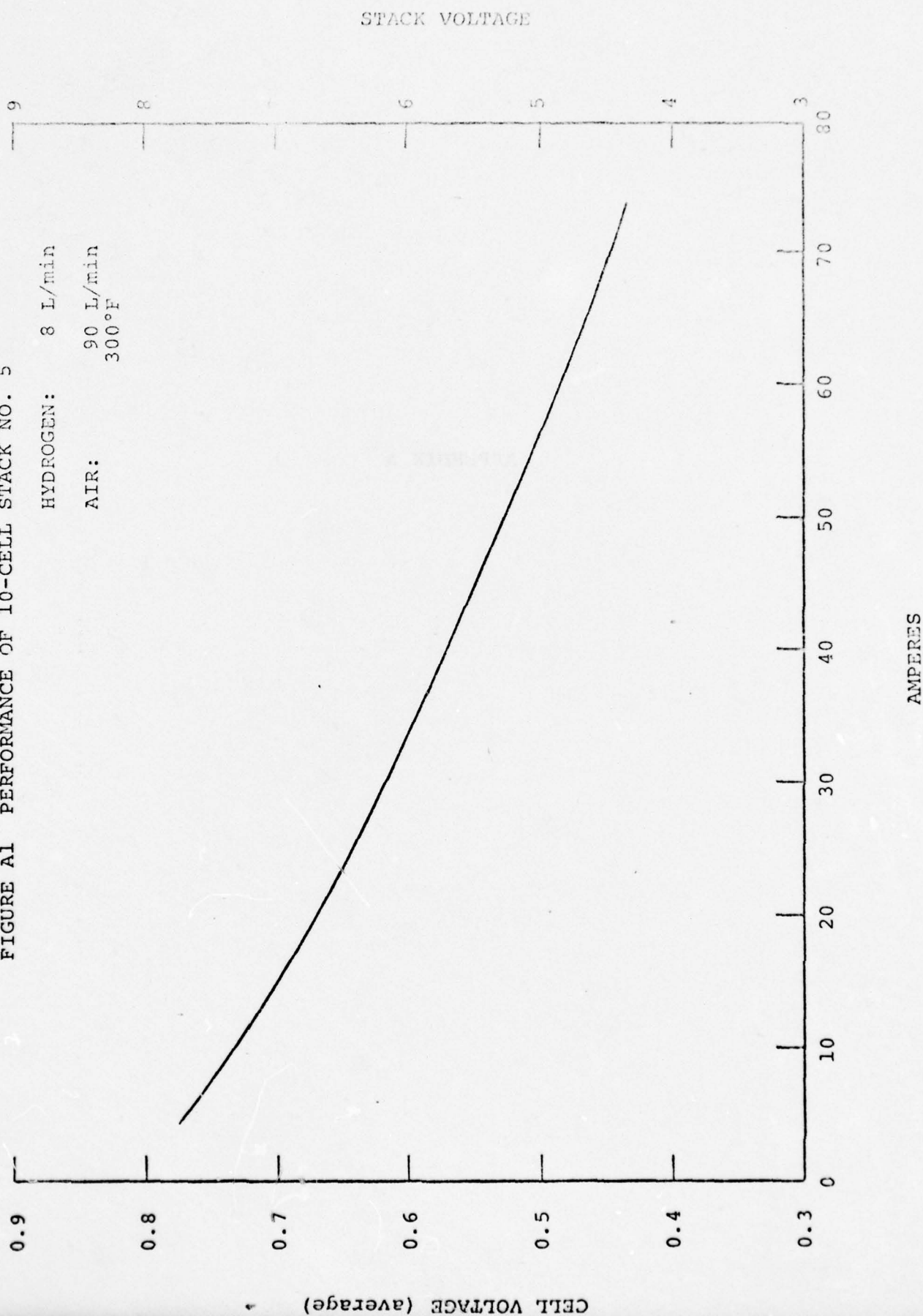
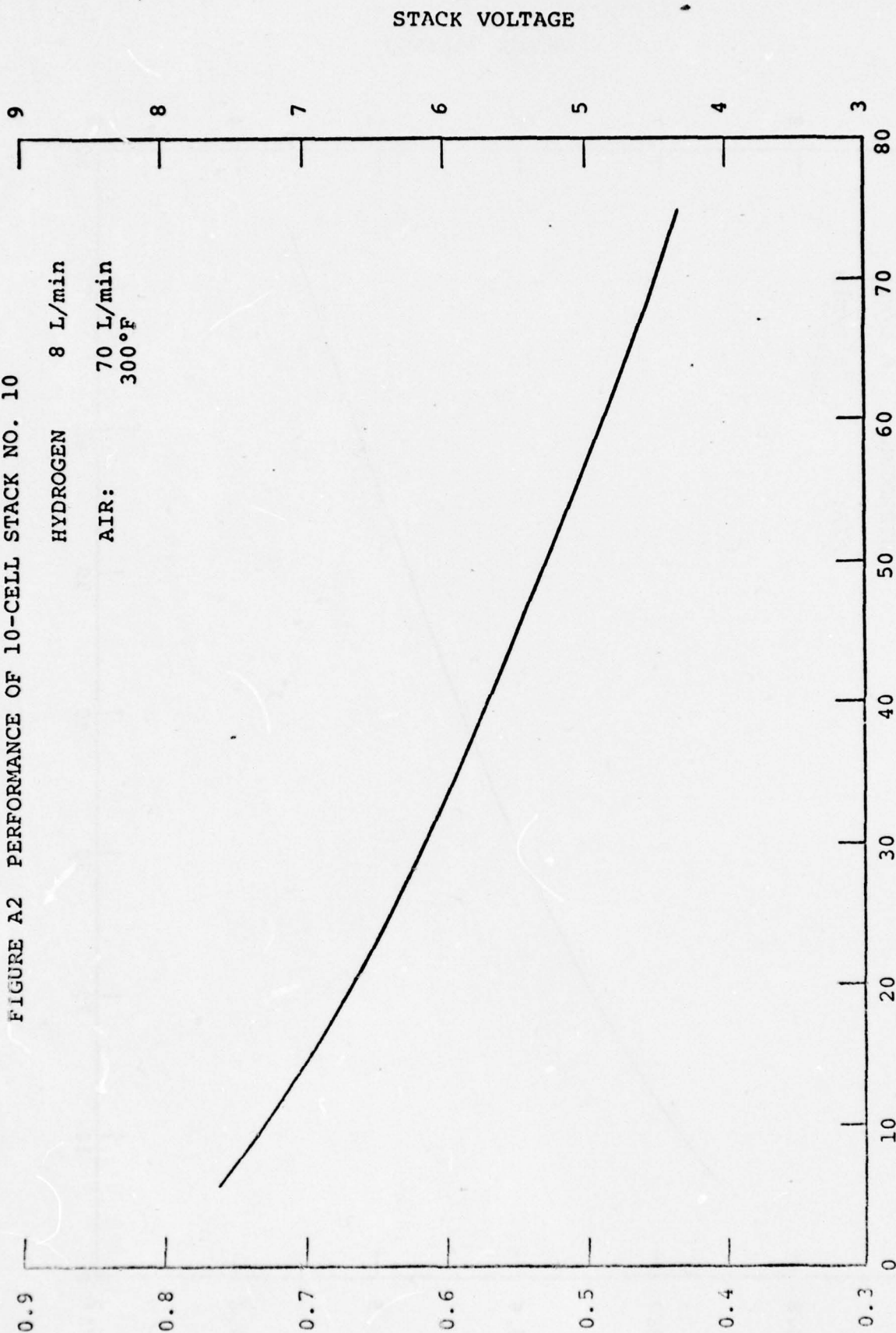
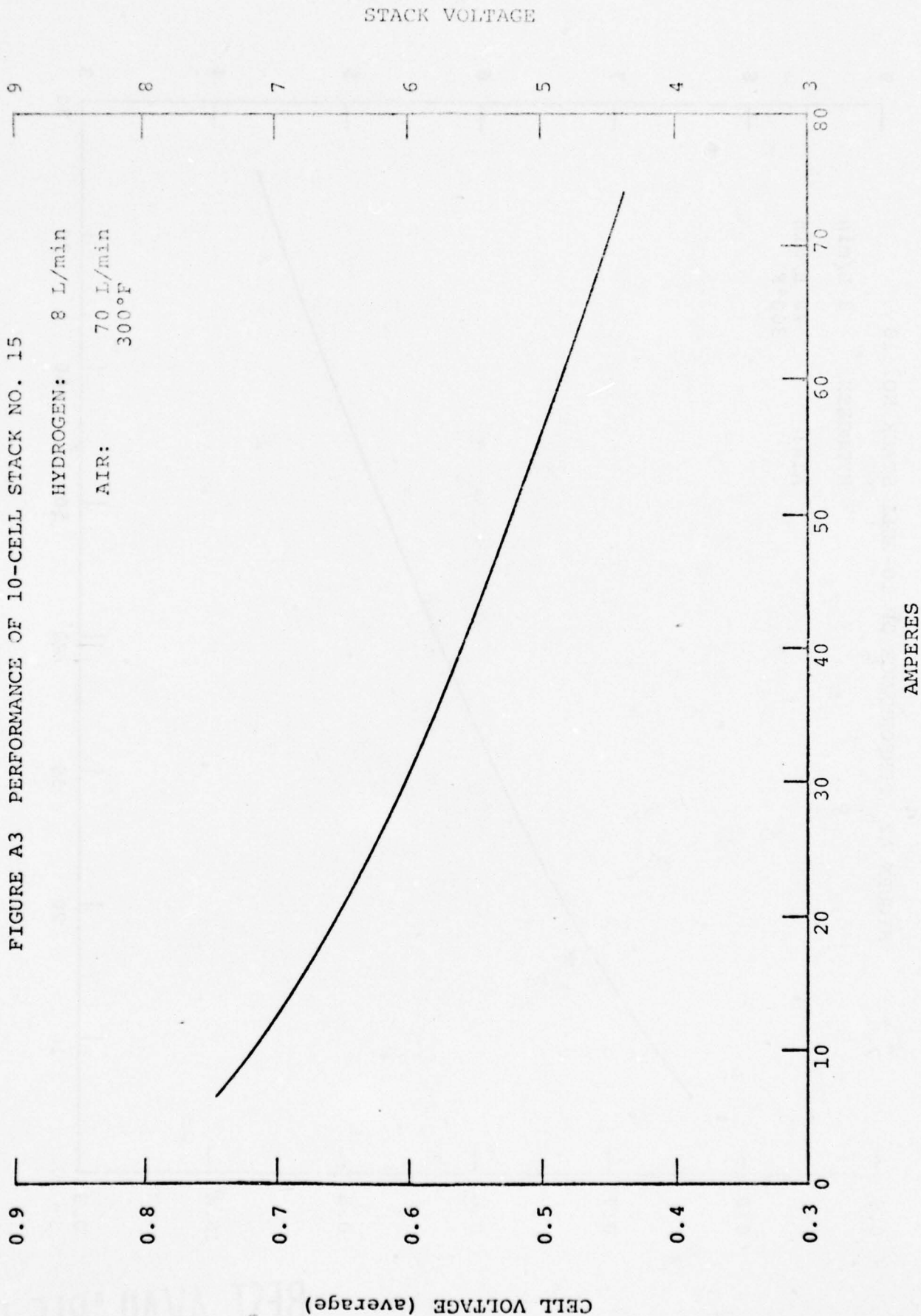


FIGURE A2 PERFORMANCE OF 10-CELL STACK NO. 10

HYDROGEN 8 L/min
AIR: 70 L/min
300°F



CELL VOLTAGE (average)



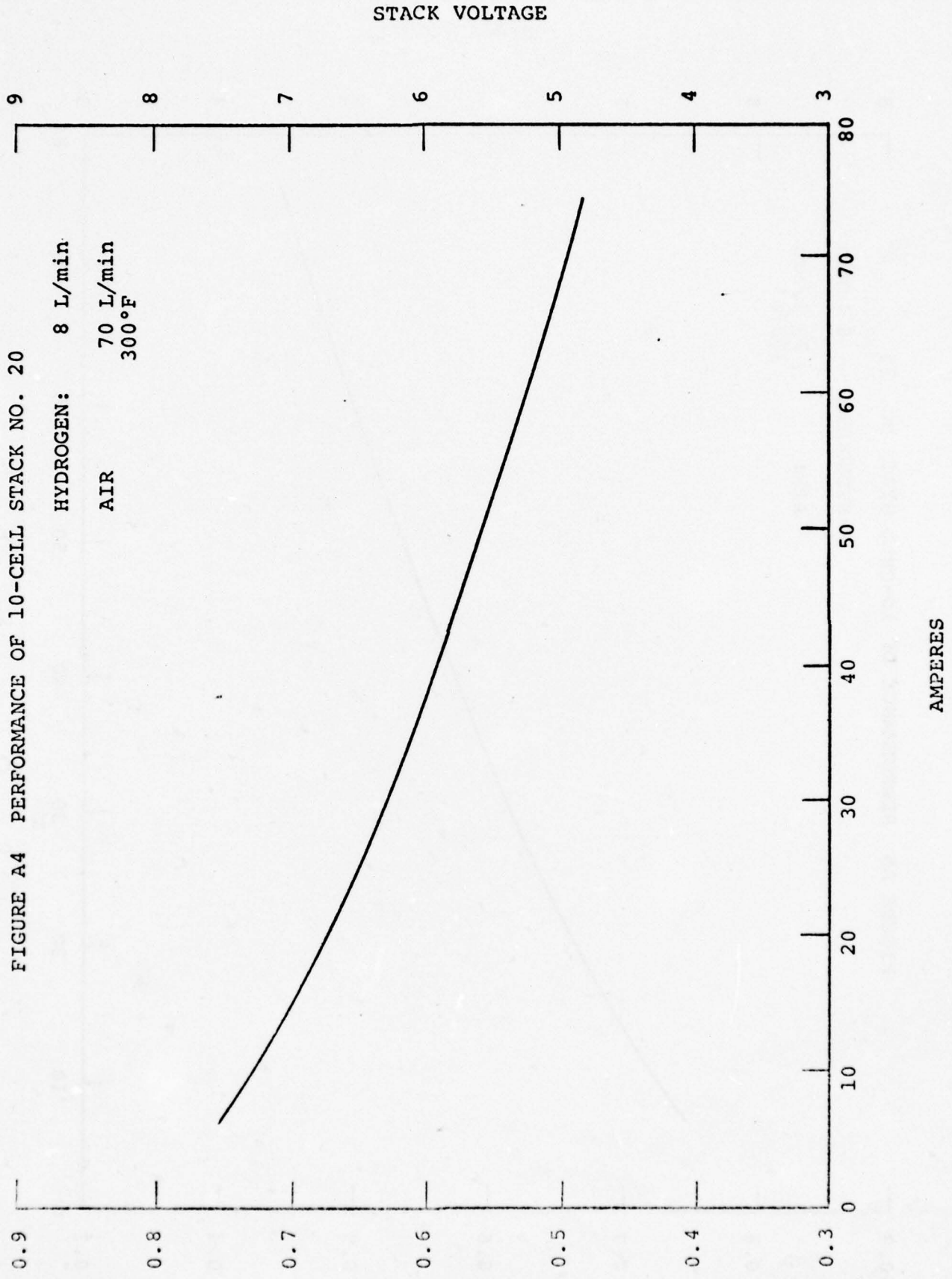


FIGURE A4 PERFORMANCE OF 10-CELL STACK NO. 20

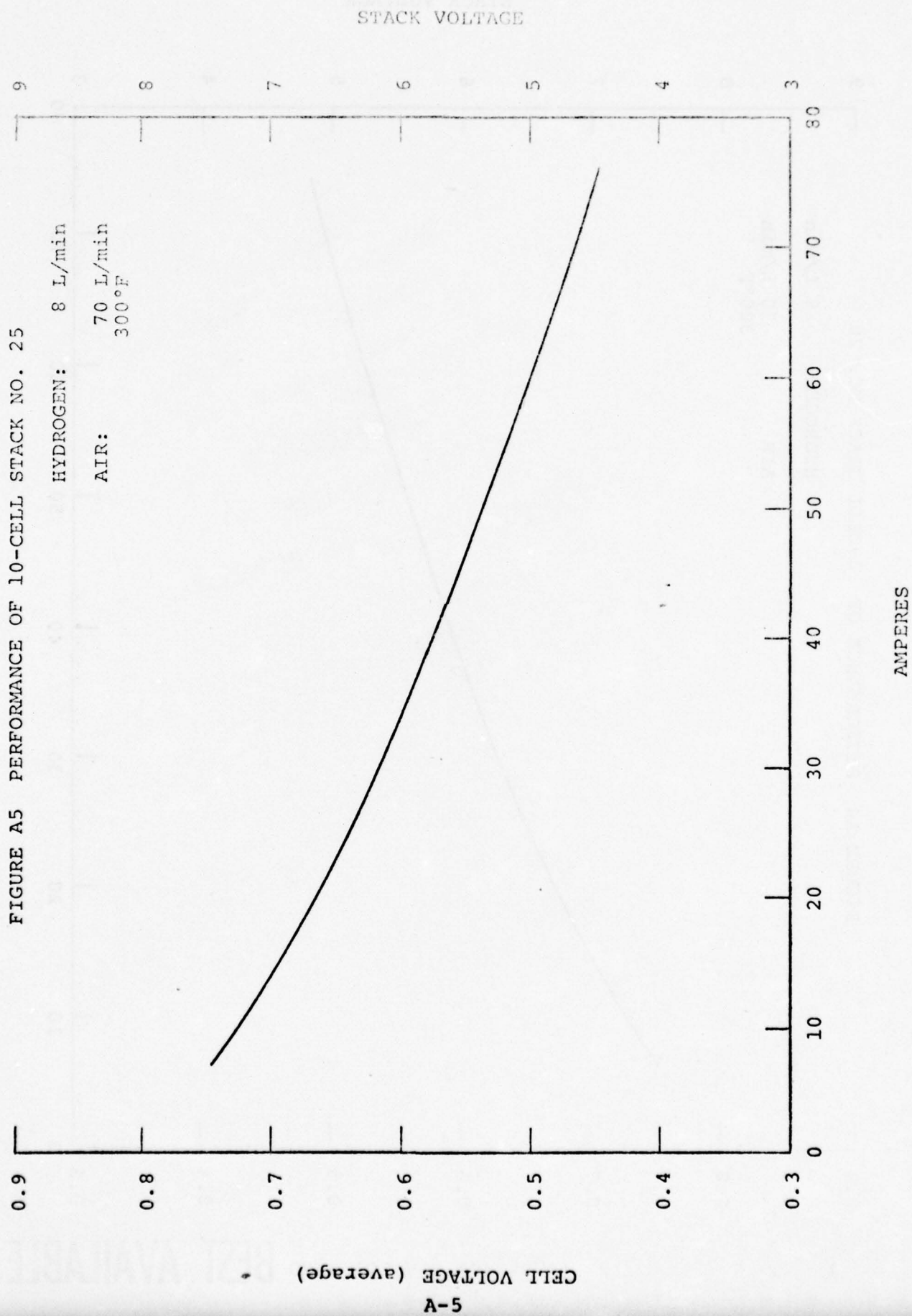
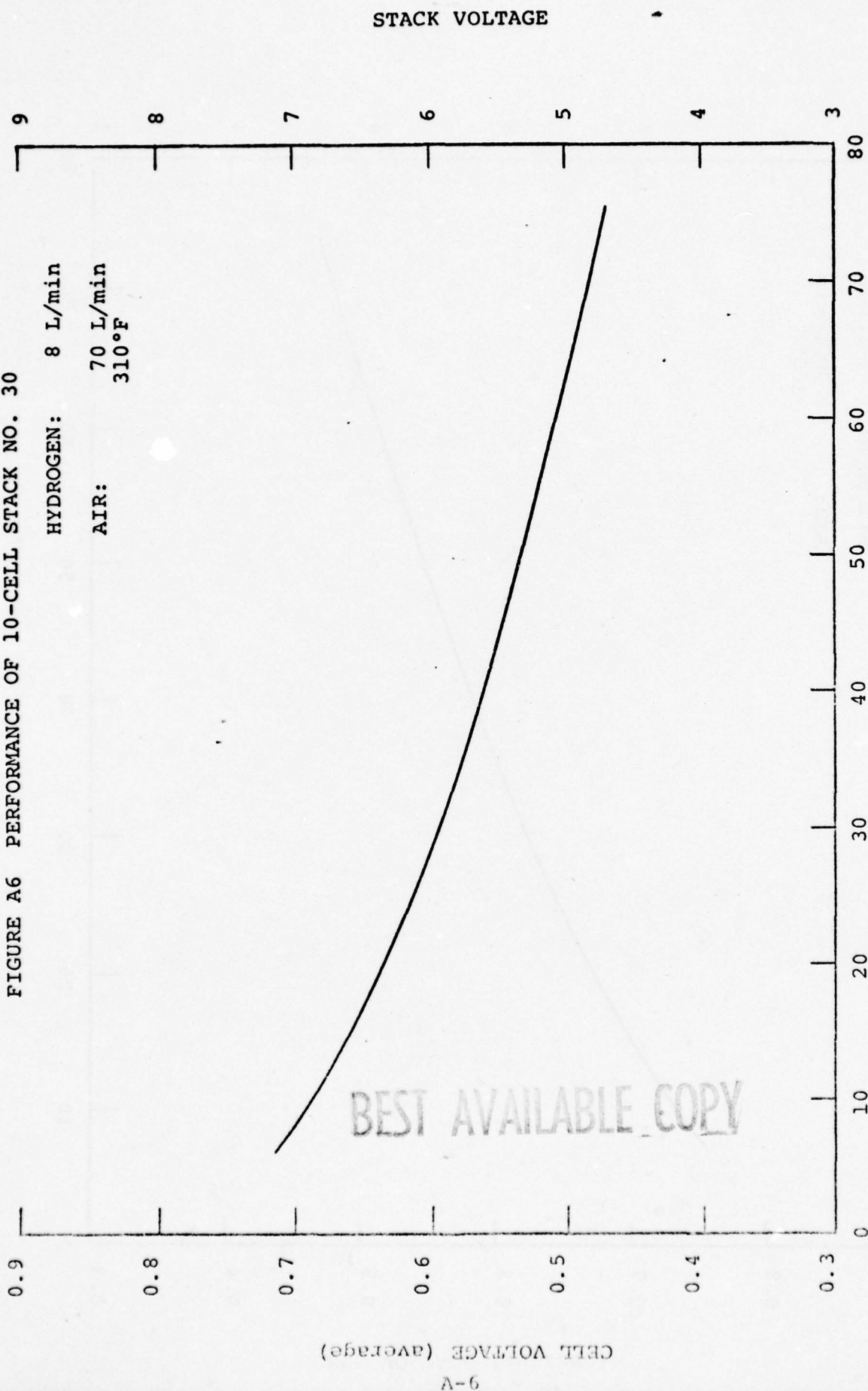


FIGURE A6 PERFORMANCE OF 10-CELL STACK NO. 30

HYDROGEN: 8 L/min

AIR: 70 L/min
310°F



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FIGURE A7 PERFORMANCE OF 10-CELL STACK NO. 35

HYDROGEN: 8 L/min

AIR: 70 L/min
300°F

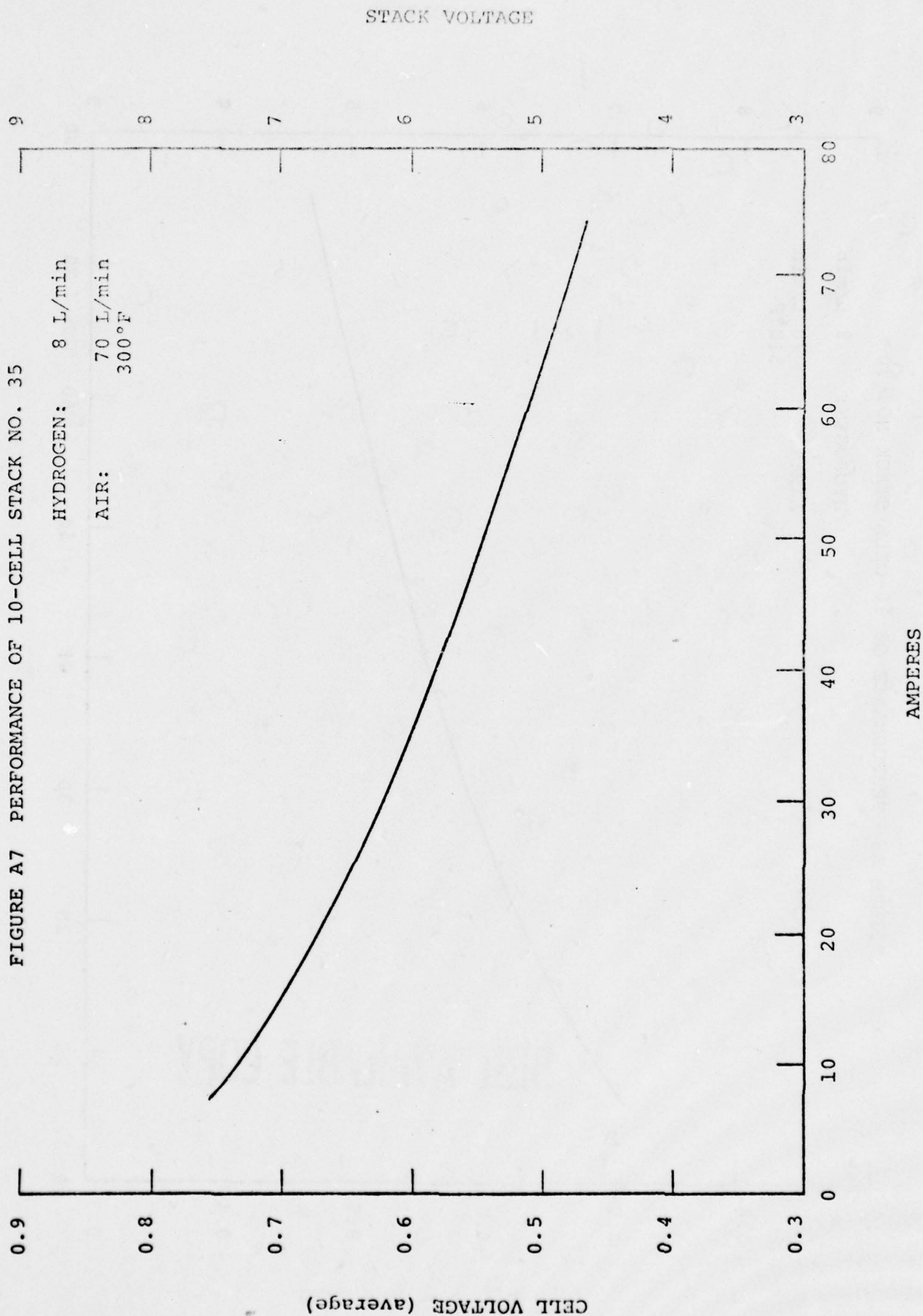


FIGURE A8 PERFORMANCE OF 10-CELL STACK NO. 40

HYDROGEN: 8 L/min

AIR: 70 L/min
320°F

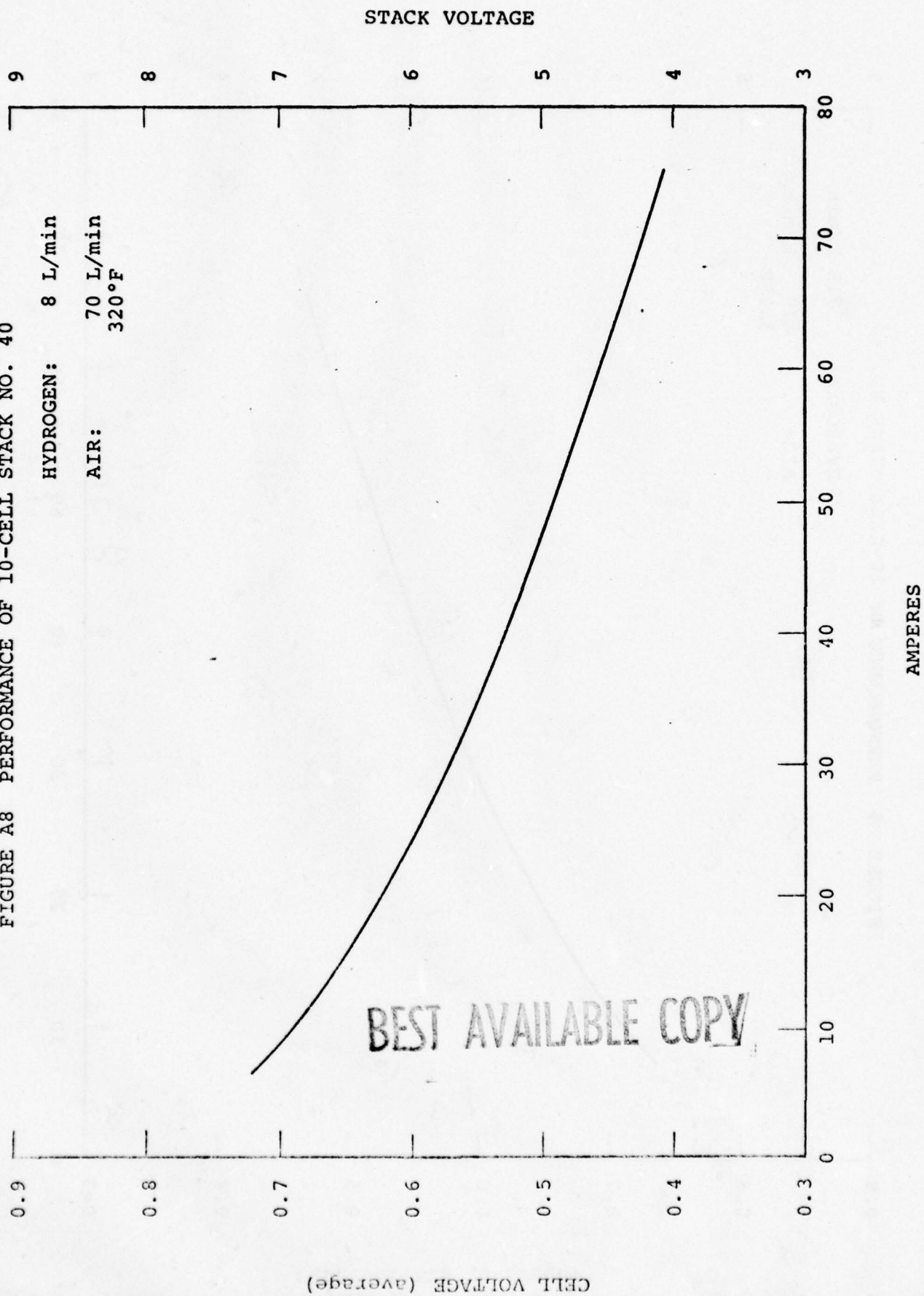


FIGURE A9 PERFORMANCE OF 10-CELL STACK NO. 45

HYDROGEN: 7.5 L/min
AIR: 70 L/min
320°F

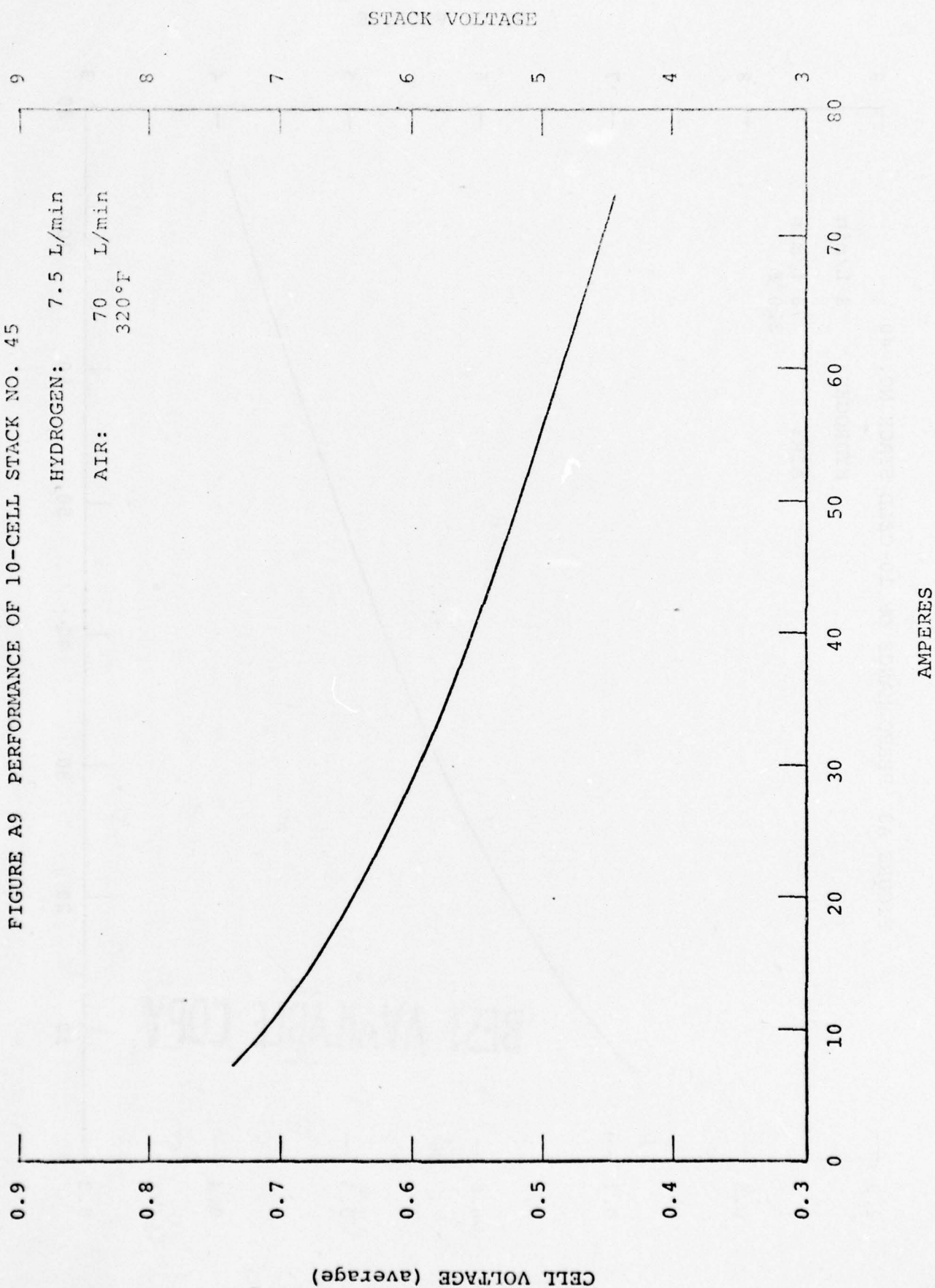
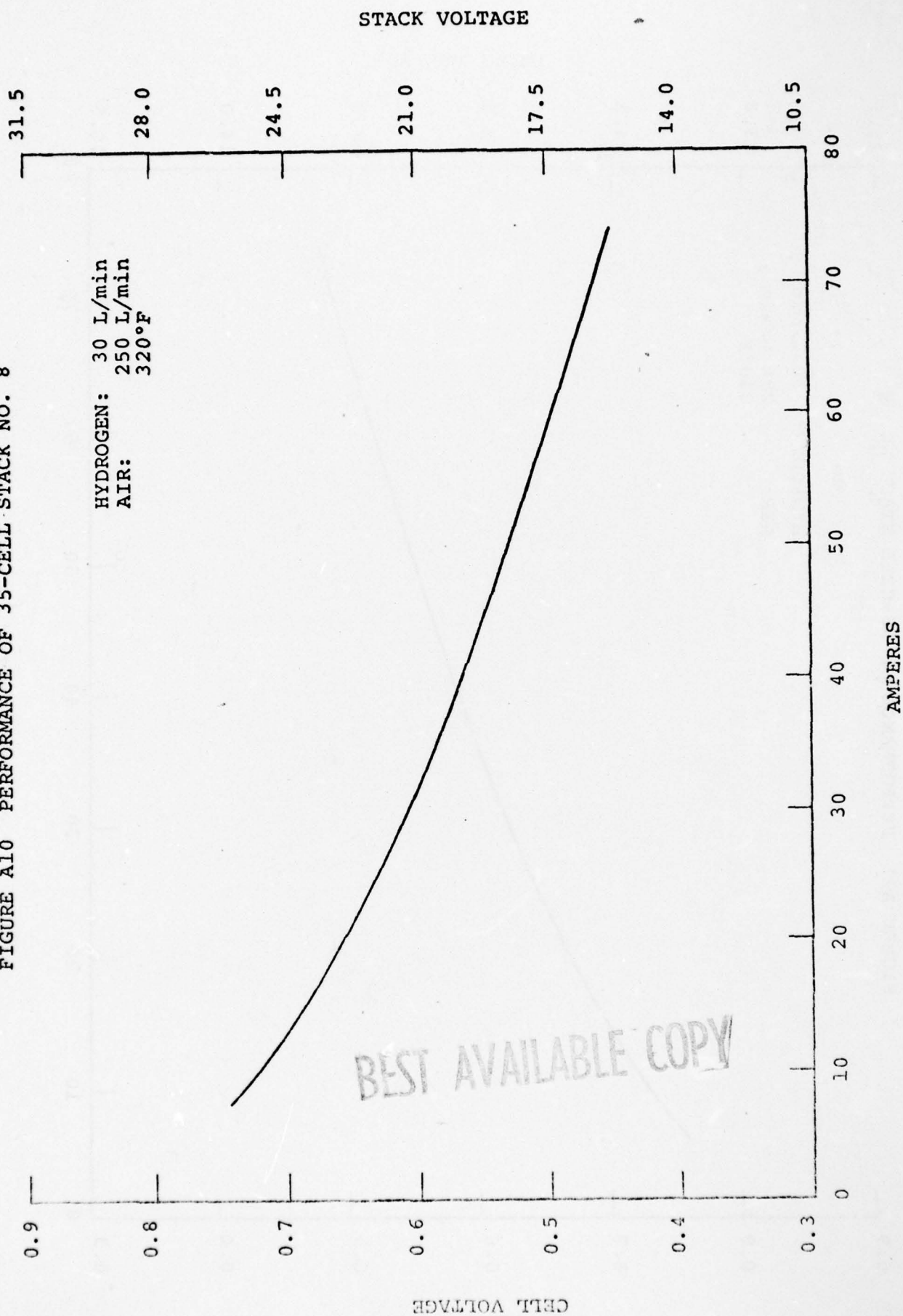


FIGURE A10 PERFORMANCE OF 35-CELL STACK NO. 8



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FIGURE A11 PERFORMANCE OF 35-CELL STACK NO. 9

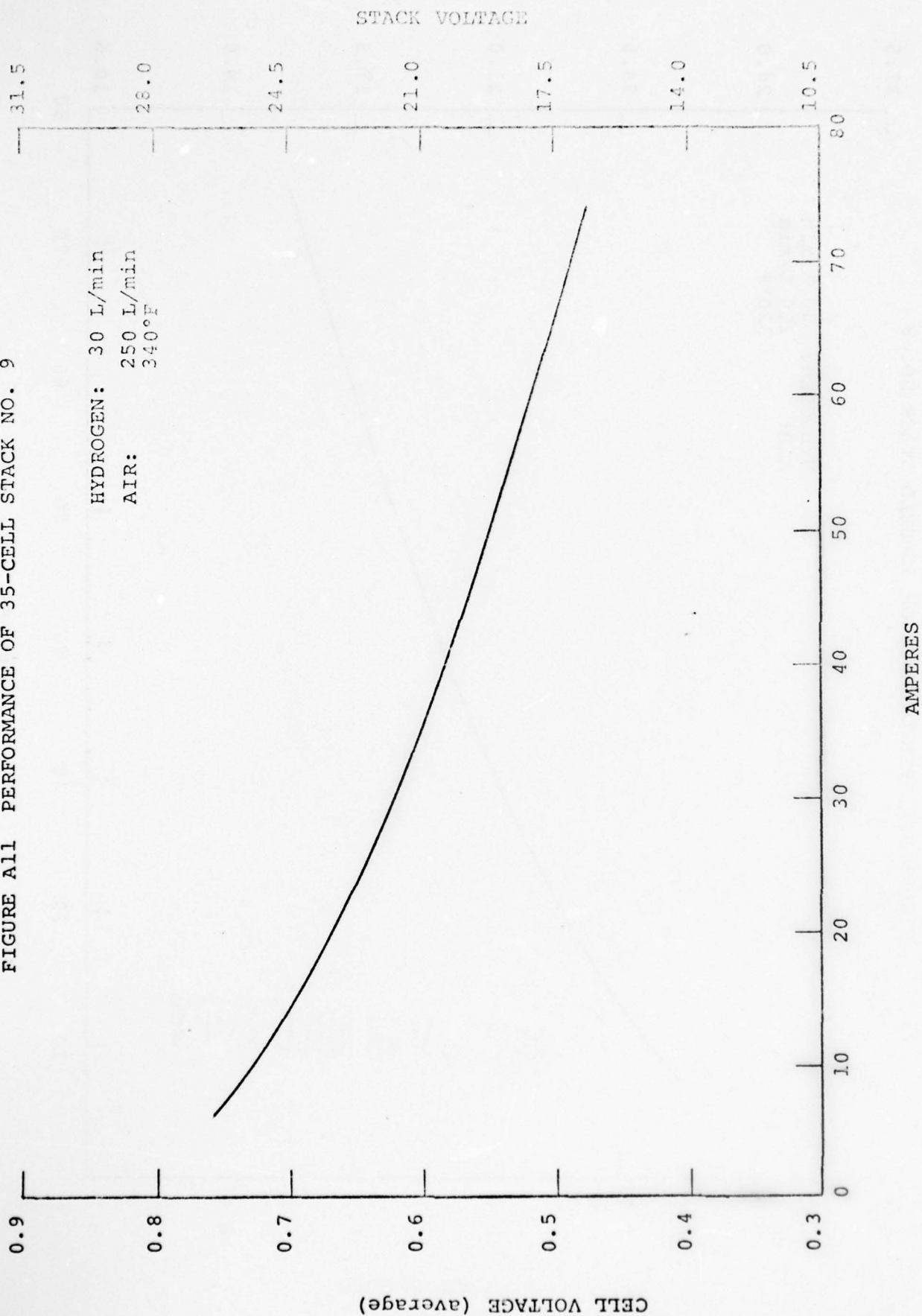
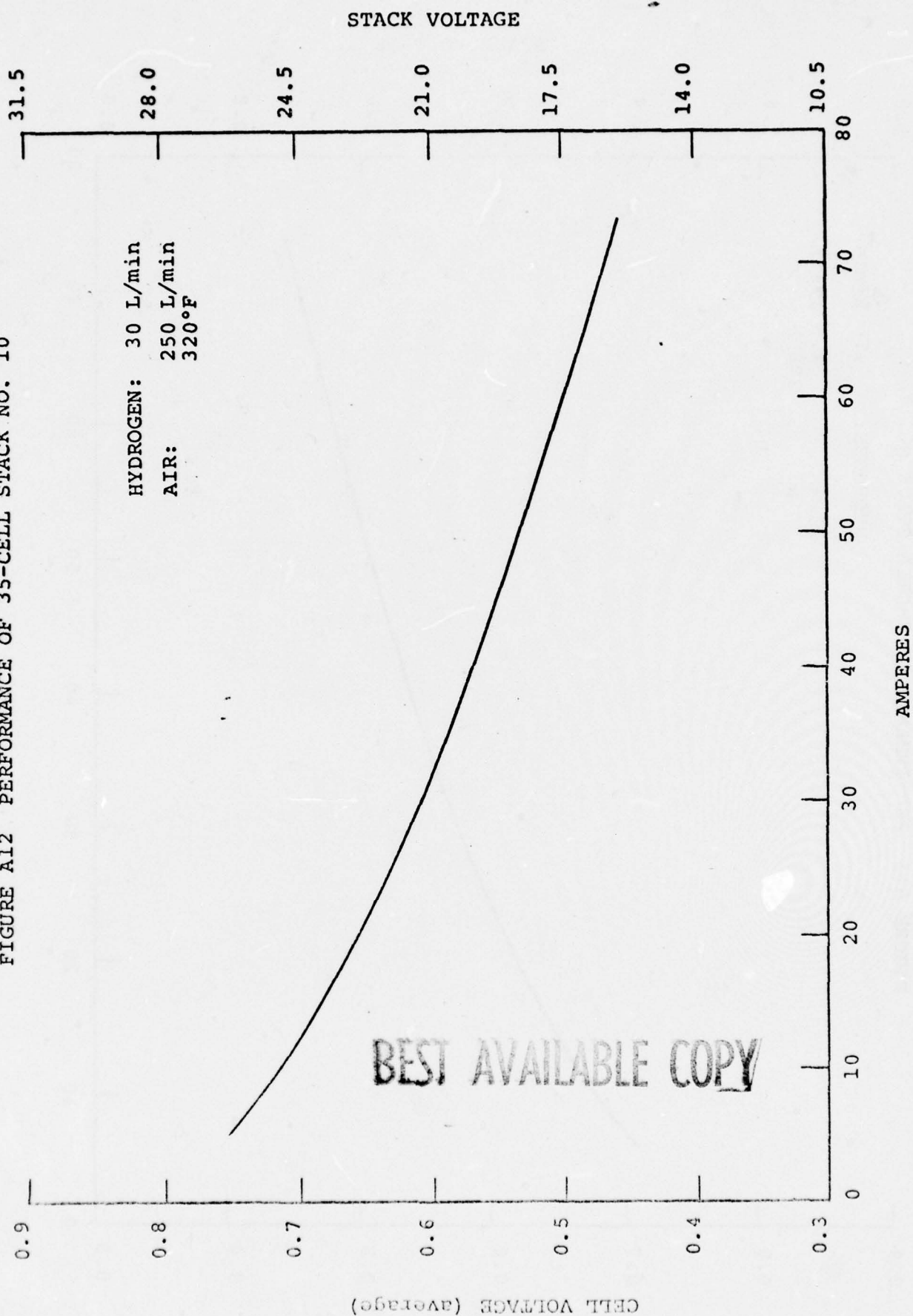


FIGURE A12 PERFORMANCE OF 35-CELL STACK NO. 10



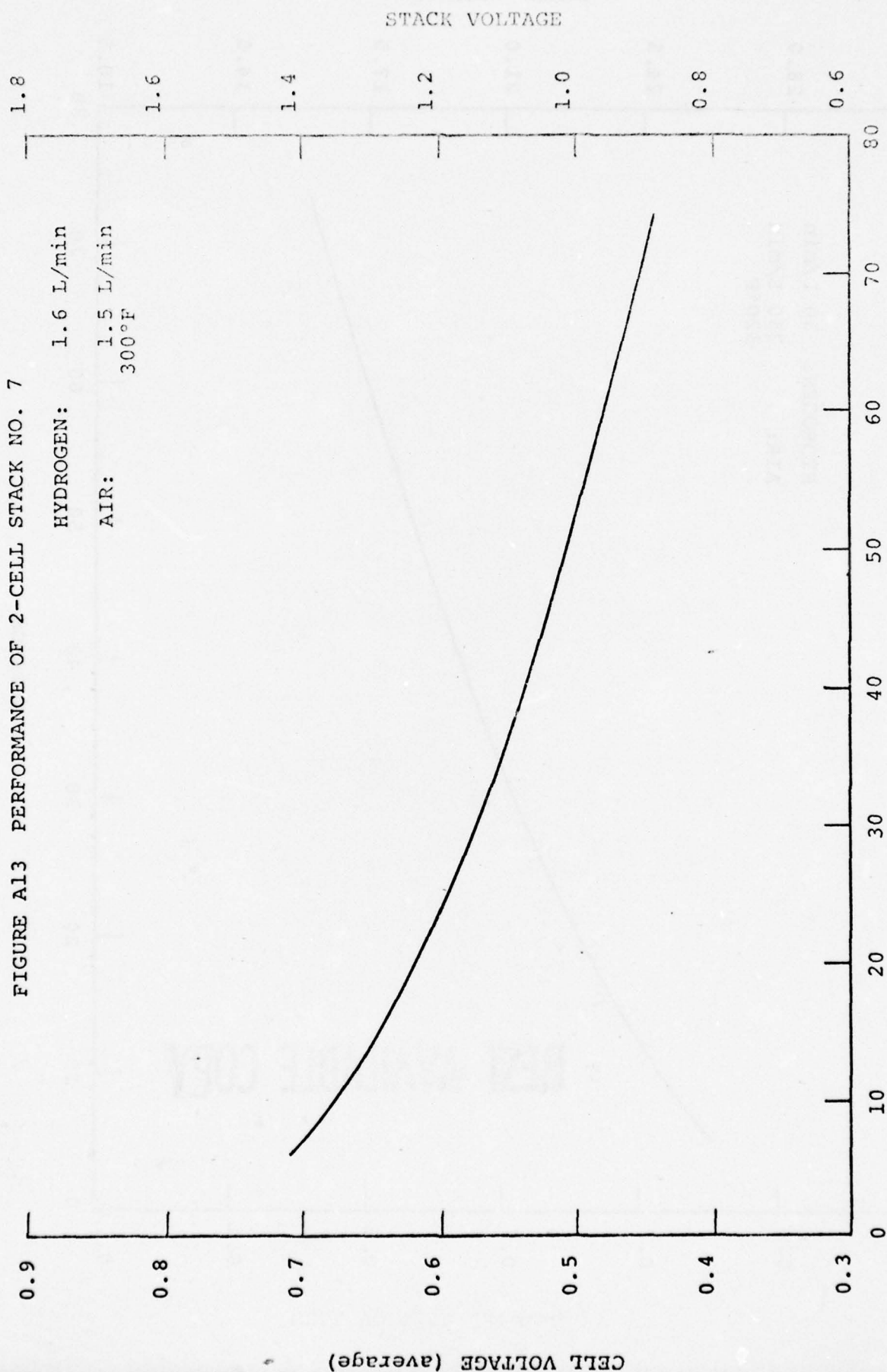
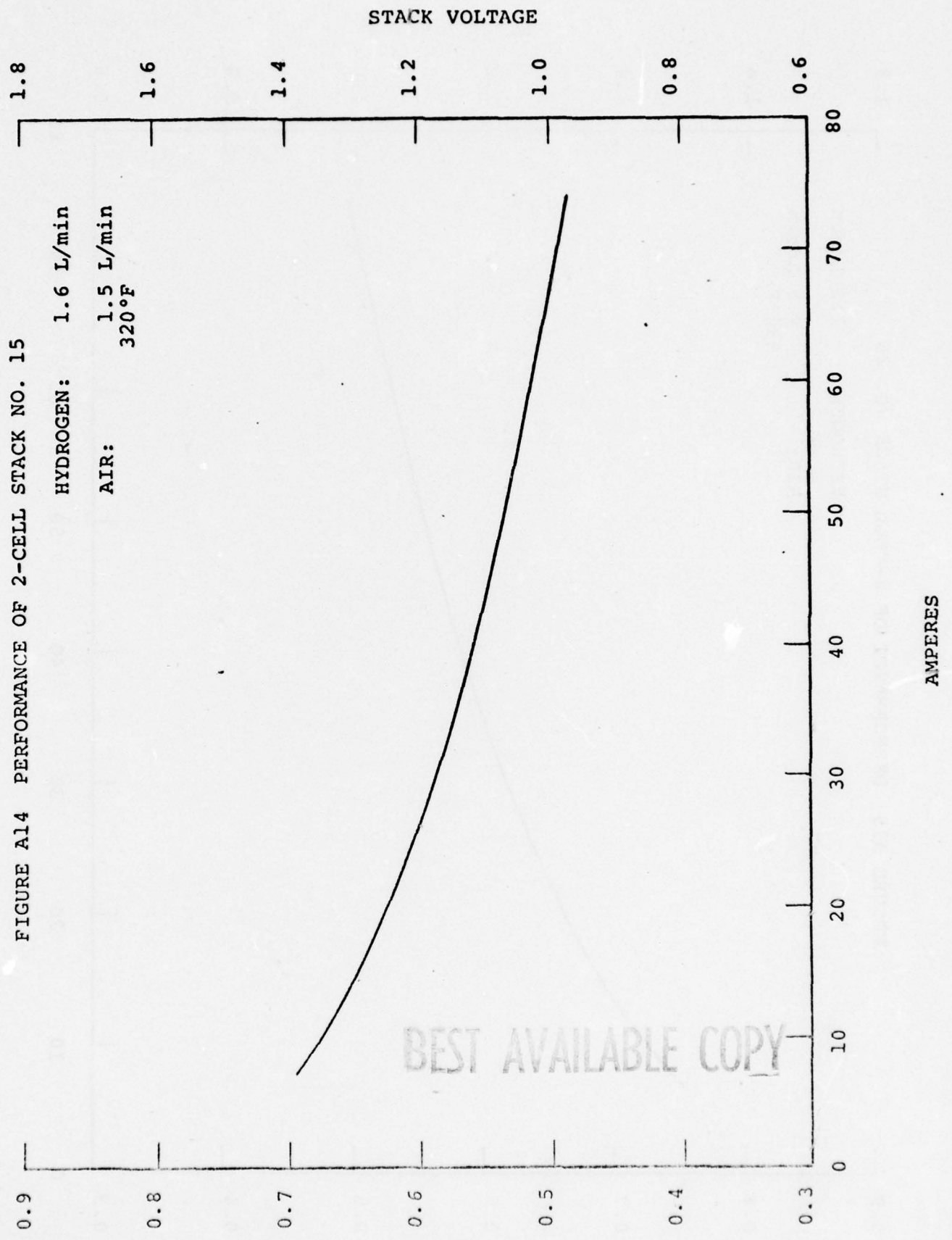


FIGURE A14 PERFORMANCE OF 2-CELL STACK NO. 15

HYDROGEN: 1.6 L/min
AIR: 1.5 L/min
320°F



CELL VOLTAGE (average)

STACK VOLTAGE

AMPERES

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FIGURE A15 PERFORMANCE OF 2-CELL STACK NO. 25

HYDROGEN: 1.5 L/min

AIR: 9.5 L/min
330°F

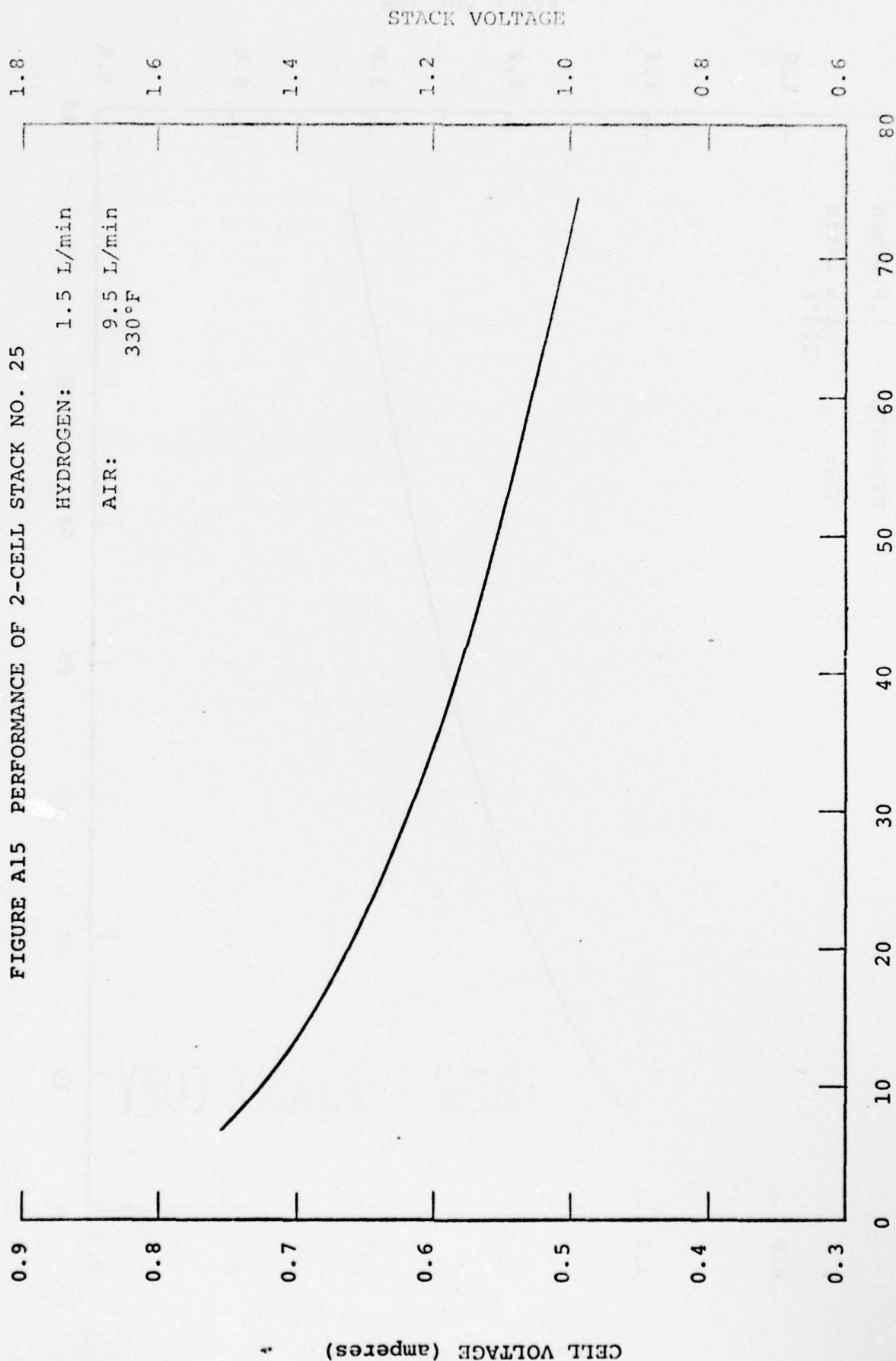
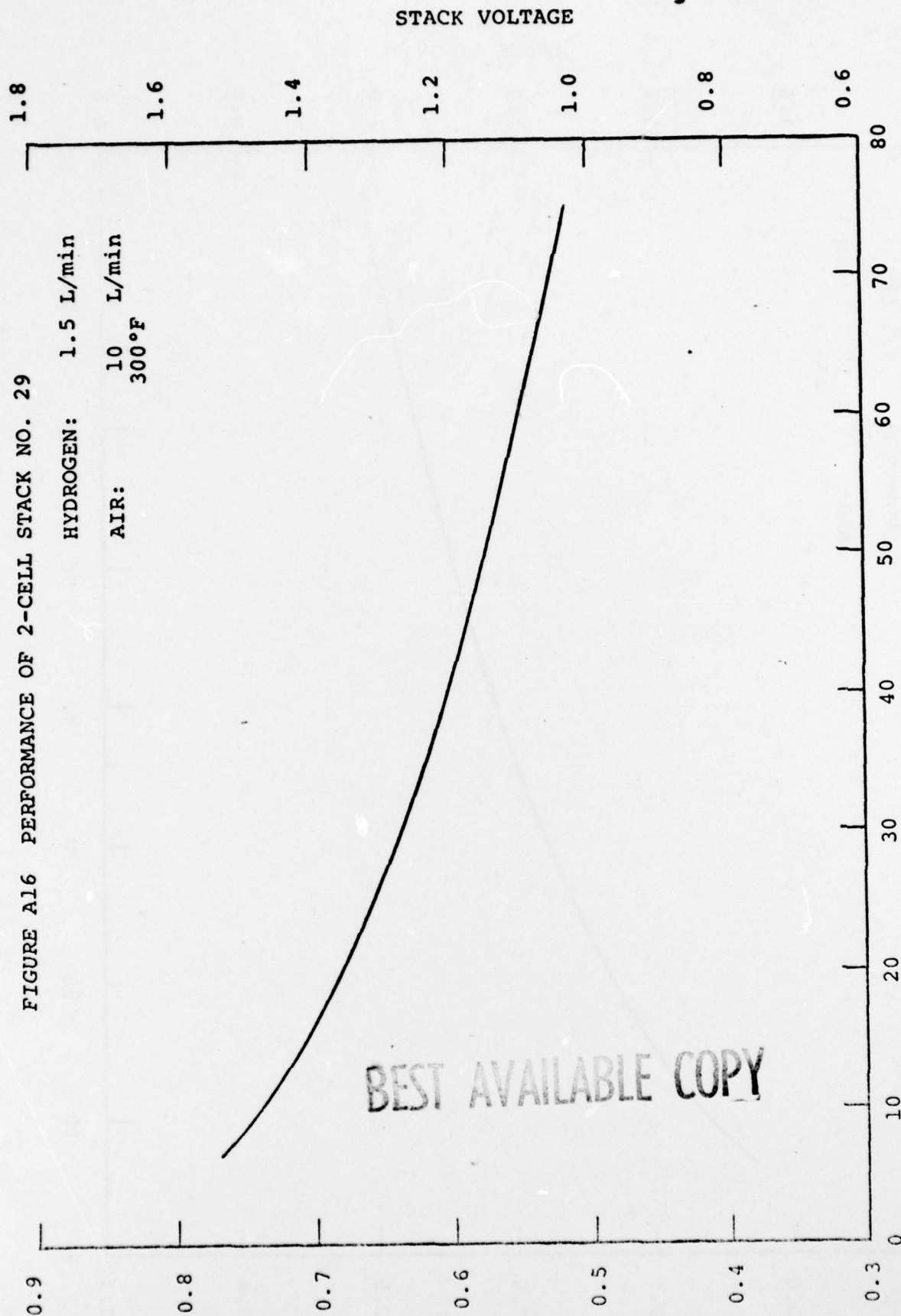


FIGURE A16 PERFORMANCE OF 2-CELL STACK NO. 29

HYDROGEN: 1.5 L/min

AIR: 10 L/min
300°F



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FIGURE A17 PERFORMANCE OF 2-CELL STACK NO. 30

HYDROGEN: 1.5 L/min
AIR: 10 L/min
335°F

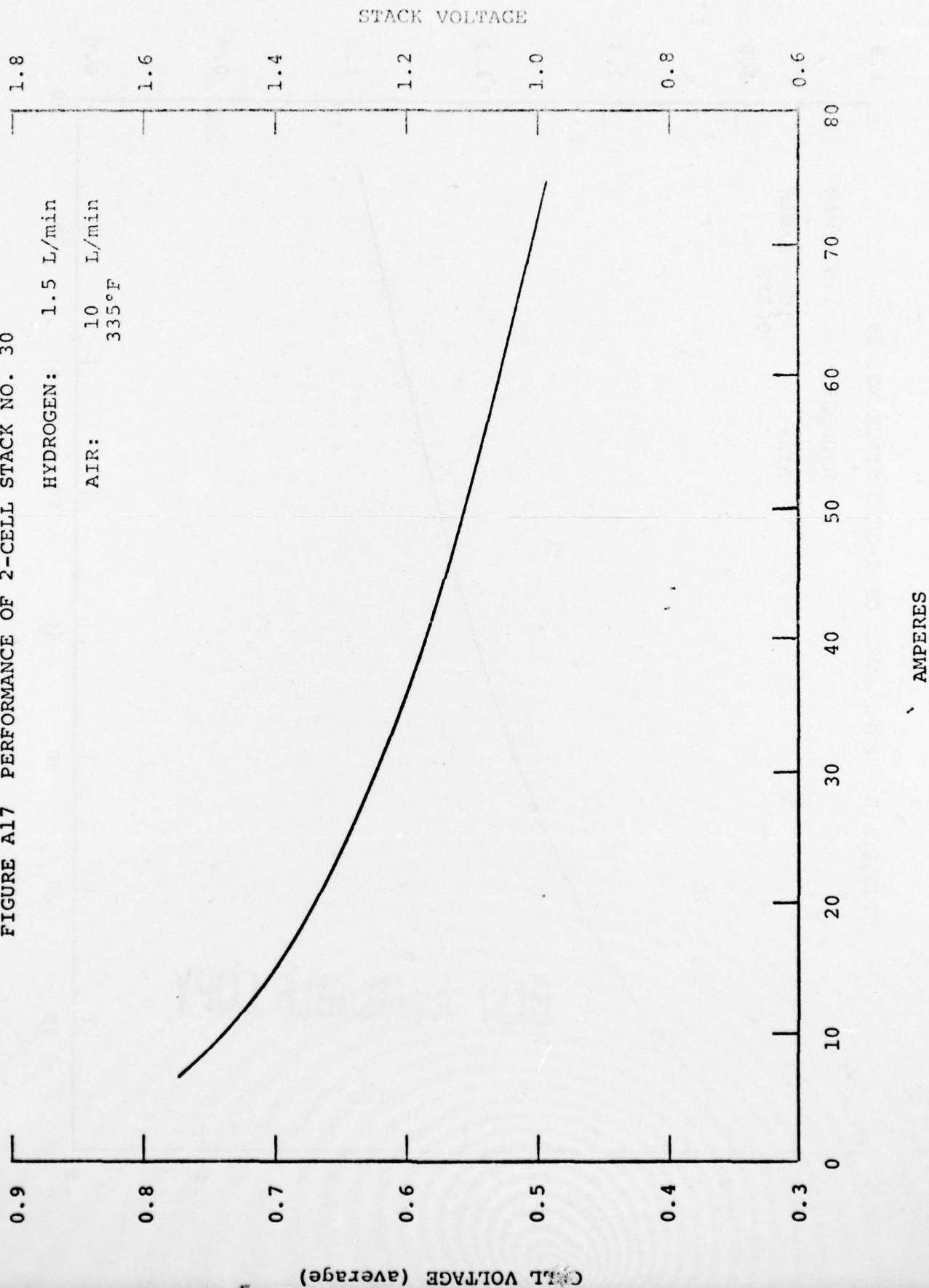


TABLE AI

DATA SUMMARY, TWO CELL STACKS

STACK NO.	BUILD NO.	PLATE RESIN %	MATRIX CODE	VOLTAGE @ 40A START	END	HOURS TESTED	NOTES
1	37	A 18	D	1.2	1.08	190	
2	42	A 20	B	1.19	1.11	530	
3	61	A 36	H	1.06	1.04	240	1 or 2
4	62	A 35	H	1.16	.98	450	1 or 2
5	63	A 33	I	1.09	.92	1430	
6	65	C 33	A	1.19	1.04	1300	
7	66	C 33	A	1.15	1.09	1100	6
8	67	C 33	I	.91	.80	220	
9	69	C 33	A	1.09	1.02	1510	
10	71	H 20	B	.97		<20	1 or 2
11	72	A 33	B	.90		<20	1 or 2
12	73	A 33	B	.96		<20	1 or 2
13	74	A 33	B	1.12	1.06	220	1 or 2
14	75	C 27	B	1.12	1.03	100	1 or 2
15	80	C 33	A	1.09	1.16	100	6
16	81	C 33	A	1.15	1.02	740	
17	86	C 33	A	1.04	1.04	70	3
18	87	C 33	H	1.05	.98	20	
19	88	C 33	B	1.07	.92	50	2
20	90	C 33	A	1.04	1.16	50	
21	91	C 33	I	1.15	1.03	530	
22	92	C 33	B	1.04	.95	520	
23	93	C 30	H	1.02	.98	2030	
24	94	C 33	I	1.09	.95	145	
25	95	C 33	I	1.19	1.14	340	6,
26	97	C 33	I	1.14	1.06	30	
27	99	C 33	I	1.07	.70	50	8
28	100	C 33	I	1.12	1.02	240	7
29	101	C 33	I	1.12	1.16	450	6,
30	102	C 33	I	1.15	1.14	320	6

TABLE AII

DATA SUMMARY, TEN CELL STACKS

STACK NO.	BUILD NO.	PLATE RESIN	%	MATRIX CODE	VOLTAGE @ START	40A END	HOURS TESTED	NOTES
1	4	H	22	I	5.0	-	<10	
2	5	H	24	I	4.5	-	<10	
3	6	H	24	I	< 4	-	-	
4	7	H	24	I	5.6	5.6	20	1
5	8	H	24	I	5.7	5.6	20	1
6	9	H	20	I	5.2	5.0	20	1, 2
7	11	H	25	I	4.1	-	-	
8	12	H	22	I	5.8	5.2	50	
9	13	H	25	I	< 4	-	-	
10	14	H	25	I	5.7			
11	15	H	25	I	5.6			
12	16	H	25	I	5.7	5.0	720	
13	17	H	25	I	5.3		<10	
14	21	H	25	I	5.8	5.0	650	
15	22	H	25	I	5.7	5.2	2200	
16	29	H	22	A	6.0	6.0	40	1
17	30	A	22	A	6.1	5.2	4000	1, 7
18	31	H	24	A	6.1	6.0	40	1
19	32	H	24	A	6.0	4.9	280	2
21	33	A	20	A	5.9	5.3	50	1
22	34	A	18	A	5.3			2
20	36	A	18	A	6.0	5.8	50	6
23	38	A	18	B	6.0	5.8	50	2
24	39	A	18	B	6.0	5.6	100	2
38	40	A	20	A	5.6	5.0	160	1
26	41	A	20	D	5.4	4.3	100	1
27	43	A	20	D	5.7			2
28	44	A	20	B	5.7	5.1	50	2
29	45	A	22	B	6.1	5.5	200	2
31	46	A	22	B	5.3	5.3	50	6

TABLE AII (Continued)

DATA SUMMARY, TEN CELL STACKS

<u>STACK NO.</u>	<u>BUILD NO.</u>	<u>PLATE RESIN</u>	<u>%</u>	<u>MATRIX CODE</u>	<u>VOLTAGE @ 40A START</u>	<u>END</u>	<u>HOURS TESTED</u>	<u>NOTES</u>
32	47	A	22	B	5.5	-	<10	1
25	48	A	22	D	5.7	5.7	200	1
30	49	A	22	B	5.7	5.7	<10	6
35	52	A	23	B	5.8	5.8	50	6
33	53	A	23	B	5.8	5.8	50	2
34	54	A	23	E	6.1	5.4	300	2,4,5
36	55	A	23	B	5.4	5.5	20	1
37	56	A	23	E	5.5	-	<10	2,3,4
38	59	A	23	D	5.3	5.5	<10	
39	64	A	33	I	5.8	5.0	280	
40	68	C	33	A	5.6	5.5	640	
41	84	C	33	H			<10	
42	85	C	33	A			<10	
43	98	C	33	I	5.6	4.9	<10	
44	105	C	33	I	5.3	-	<10	2,3,4
45	104	C	33	I	5.7	5.6	12	

TABLE AIII
DATA SUMMARY, 35 CELL STACKS

<u>STACK NO.</u>	<u>BUILD NO.</u>	<u>PLATE RESIN</u>	<u>%</u>	<u>MATRIX CODE</u>	<u>VOLTAGE @ 40A START</u>	<u>END</u>	<u>HOURS TESTED</u>	<u>NOTES</u>
1	50	A	22	D	21.0	19.1	20	1,2
2	51	A	23	D	19.6		10	2
3	57	A	23	D	19.9		<10	1,2
4	58	A	24	D	20.5		<10	3
5	60	A	24	D	20.5		<10	3
6	82	C	33	H			<10	
7	83	C	33	H			<10	
8	89	C	33	A	20.0		<10	6
9	96	C	33	I	20.5		<10	6
10	103	C	33	I	20.2		<10	6

NOTES TO TABLES AI - AIII

1. Fuel manifold leak
2. Gas crossover
3. Cracked bipolar plate
4. Honeycomb endplates used
5. Operated with cathode air recirculation
6. Stack delivered to MERADCOM

PLATE RESIN CODE

H = H-Resin
A = Arofene 889
C = Colloid 8440

APPENDIX B

PROJECTED STACK MANUFACTURING

As part of the contractual effort, a facility for the production of 100 1.0 KW stacks per month was designed, and equipment and manpower requirements were estimated. The component manufacturing processes developed on this program were used as the basis for the manufacturing facility. No basic modifications to the process equipment were assumed, although a double cavity mold for bipolar plate production is proposed. The 1.0 KW stack is assumed to be made with 40 5 X 15 in. cells delivering 25 watts/cell.

EQUIPMENT AND MANPOWER

The facility layout as well as the equipment and daily manhour estimates are shown in Figure B1. The critical (output-limiting) operations for each component manufacturing process are the following:

1. Electrode Process

Operation: Catalyst layer rolling
Monthly Requirement: 8,000 layers
Expected Scrap Rate: Under 10%
Equipment: Two-roll mills (3),
 18" roll width
Batch Size: Sheet size 16" X 48" cut to
 9 5" X 15" layers
Mill Process Time: 30 minutes/batch
Monthly Capacity: 9,288 layers

Note: Catalyst layer output presented in Table II of this report is based of 50% duty schedule for the mill, while in the 100 stack/mo. facility a 100% duty schedule is assumed.

2. Matrix Process

Operation: Sheet molding
Monthly Requirement: 4,000 matrices
Expected Scrap Rate: under 5%
Equipment: Sheet molds (2)
 Size 18 X 18 in.
Batch Size: 18" X 18" sheet, cut
 to 3 matrices 5" X 15"
Molding Cycle: 15 minutes/batch
Monthly Capacity: 4,128 matrices

3. Bipolar Plate Process

Operation: Plate molding
Monthly Requirement: 4,100 plates
Expected Scrap Rate: 25%
Equipment: Twin-cavity presses (2),
operated 16 hours/day
Molding Cycle: 15 minutes
Monthly Capacity: 5,504 plates

MATERIAL COST

Material cost breakdown for the present stack design is shown in Table B1. The figures are based on a power level of 25 watts/cell, or 62 w/sq. ft.

TABLE B1

STACK MATERIAL COSTS

<u>MATERIAL</u>	<u>PRICE</u>	<u>USAGE/CELL</u>	<u>COST, \$/CELL</u>	<u>COST, \$/KW</u>
Pt, Rh	\$ 7/g	1.6g	11.20	448
Support	\$ 1/sq. ft.	1 sq. ft.	1.00	40
Kynol	\$15/lb	0.03 lb	0.45	18
Arofene Resin	\$ 3/lb	0.2 lb	0.60	24
Graphite	\$ 1/lb	0.5 lb	0.50	20
Miscellaneous (TFE, Resinox, etc.)			.25	10
			<hr/> 14.00	<hr/> 560

We see from this table that 80% of the stack material cost resides with the catalyst. The major material cost reduction will, therefore, come from catalyst reduction. Such reduction may be possible thru the use of supported catalysts (Kocite or Pt on carbon). A 75% reduction of catalyst cost would yield materials cost of \$225/KW.

Endplates, manifolds, and other external hardware are not included in these estimates. The cost for these items will depend on the size of the stack and on application, i.e., stationary stacks can probably be built with low cost, heavy steel endplates, while portable systems will require development of more expensive, lightweight hardware. Also, for the purposes of this calculation, cost of scrap material is neglected (noble metal scrap is normally recoverable).

LABOR COSTS

Based on the manpower estimates summarized for this process in Figure B1, direct labor costs can be calculated* to be about \$315/KW. This figure would, of course, be reduced in a facility where a higher production rate would justify installation of continuous process equipment for the electrode and for the matrix. In the 100 stacks/month facility shown in Figure B1, 200 manhours/day are estimated in the batch-type matrix and electrode processes. Assuming a 2/3 reduction in labor for these processes in going to a continuous process, a reduction in direct labor costs to \$183/KW would result.

STACK WEIGHT

A cell component weight breakdown is shown in Table B2. Endplate and manifold weights are not included in this analysis, since the weight contribution of these components depends on the number of cells in the stack, endplate design, etc.

Table B2 Cell Component Weights

<u>Component</u>	<u>Weight, g/cell</u>
Bipolar plate	215
Anode	8
Cathode	9
Matrix	9
Electrolyte	18
Ta Shims	2
Cement	2
<hr/>	
Total	263 g (0.58 lb)

Since 40 cells are required for a 1.0 KW stack, the stack weight becomes 23.2 lbs/KW, not including endplates and manifolds.

About 82% of the stack weight is made up of bipolar plates; therefore, a major weight reduction would result if lighter plates could be used. A 20% weight reduction in this component would reduce the stack weight to 19.4 lbs/KW.

* (368 hours/day X 21.5 days/month X \$4/hr)/100 stacks/month

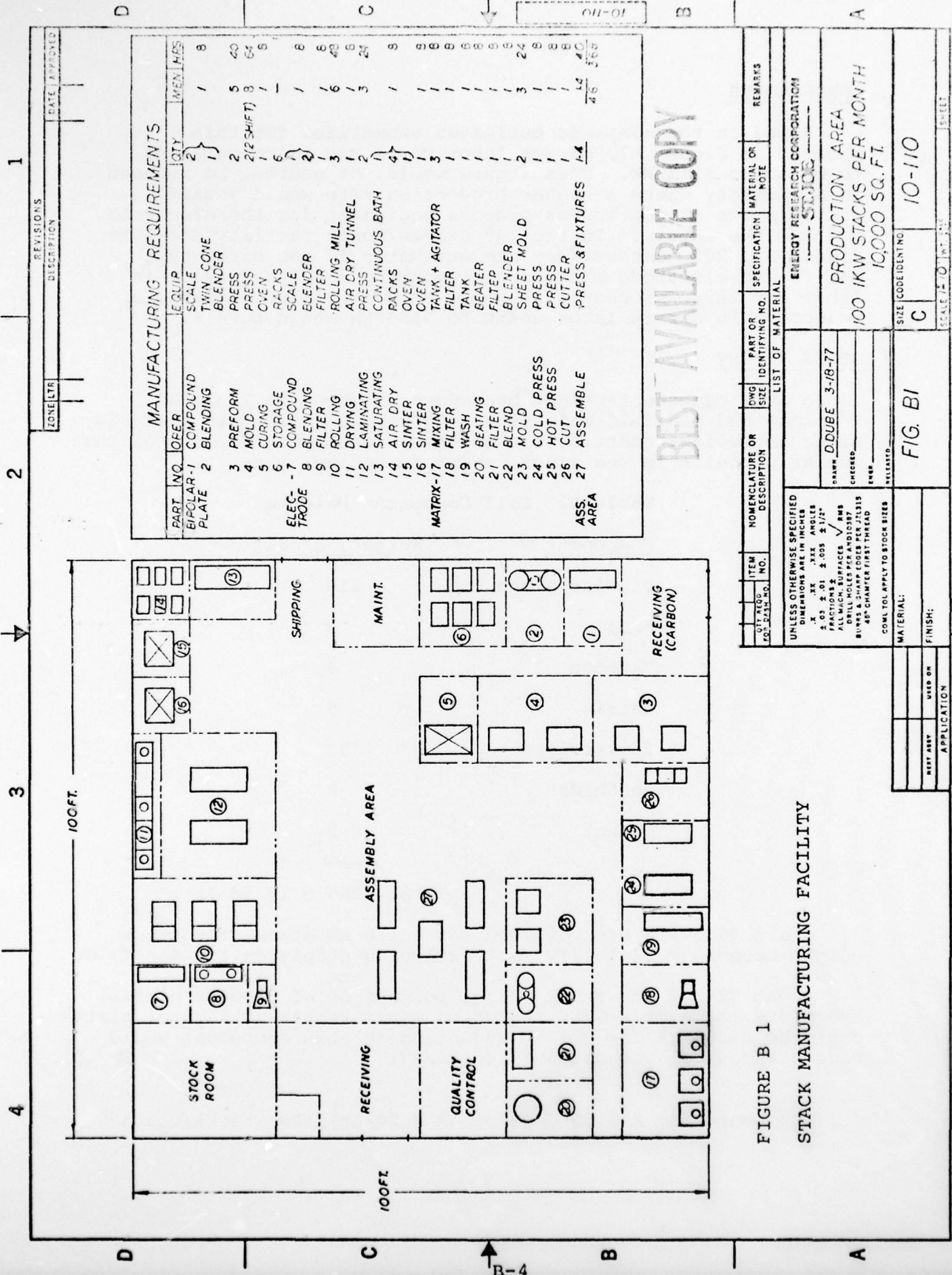


FIGURE B 1

STACK MANUFACTURING FACILITY

ITEM NO.		NOMENCLATURE OR DESCRIPTION		DWG. NO. OR IDENTIFYING NO.		PART OR SPECIFICATION		MATERIAL OR NOTE		REMARKS	
QTY REQD EQT. 5213 NO.		LIST OF MATERIAL									
UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES											
.X .XX .XXX ANGLES											
2.03 2.01 2.005 2 1/2"											
FRACTIONS 2 SURFACES ✓ RMS											
DRILL HOLES PER ANSI B91.1											
BLUNTS & SHARP EDGES PER J71315											
45° CHAMFER FIRST THREAD											
COML TOL. APPLY TO STOCK SIZES											
MATERIAL:				FIG. B1		SIZE CODE IDENT NO.		C		10-110	
FINISH:											
ENERGY RESEARCH CORPORATION											
STOCK											
PRODUCTION AREA											
100 KW STACKS PER MONTH											
10,000 SQ. FT.											
SCALE 1/4" = 10' INT. 1/2"											
SHEET											

MANUFACTURING REQUIREMENTS				
PART NO.	QTY	EQUIP	QTY	MEN HRS
BIPOLAR-1 COMPOUND	2	SCALE	1	8
PLATE 2 BLENDING	1	TWIN CONE BLENDER	1	40
3 PREFORM	2	PRESS	2	5
4 MOLD	2	PRESS	2	3
5 CURING	2	OVEN	2	64
6 STORAGE	6	RACKS	6	5
ELEC-7 COMPOUND	1	SCALE	1	8
8 BLENDING	2	BLENDER	2	1
9 FILTER	1	FILTER	1	6
10 ROLLING	3	ROLLING MILL	3	42
11 DRYING	1	AIR DRY TUNNEL	1	8
12 LAMINATING	2	PRESS	2	3
13 SATURATING	1	CONTINUOUS BATH	1	24
14 AIR DRY	4	PACKS	4	1
15 SINTER	1	OVEN	1	9
16 SINTER	1	OVEN	1	9
MATRIX-17 MIXING	3	TANK + AGITATOR	3	1
18 FILTER	1	FILTER	1	1
19 WASH	1	TANK	1	1
20 BEATING	1	BEATER	1	1
21 FILTER	1	FILTER	1	1
22 BLEND	1	BLENDER	1	1
23 MOLD	2	SHEET MOLD	2	3
24 COLD PRESS	1	PRESS	1	24
25 HOT PRESS	1	PRESS	1	1
26 CUT	1	CUTTER	1	1
27 ASSEMBLE	14	PRESS & FIXTURES	14	40
ASS. AREA	46		46	365

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